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AN EXPERIMENTAL INVESTIGATION IN THE
BEHAVIOR OF METALLIZED SOLID PROPELLANTS

by

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A reduced smoke ZrC propellant and two propellants with low aluminum loadings were utilized. ZrC was observed to agglomerate and ignite on the propellant surface before being ejected. The aluminum did not agglomerate but did ignite on the propellant surface. ZrC was found to burn in part with a detached flame and the flame moved closer to the particle surface as pressure increased. Aluminum particles were observed to burn with similar behavior, but with flames more detached from the particle surface. Increased aluminum loading resulted in smaller particles above the propellant surface, but the flames were further from the particle surfaces.

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An Experimental Investigation of the Behavior of Metallized Solid Propellants

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ABSTRACT

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A reduced smoke ZrC propellant and two propellants with low aluminum loadings were utilized. ZrC was observed to agglomerate and ignite on the propellant surface before being ejected. The aluminum did not agglomerate but did ignite on the propellant surface. ZrC was found to burn in part with a detached flame and the flame moved closer to the particle surface as pressure increased. Aluminum particles were observed to burn with similar behavior, but with flames more detached from the particle surface. Increased aluminum loading resulted in smaller particles above the propellant surface, but the flames were further from the particle surfaces.

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I. INTRODUCTION

Missiles play an integral role in the defense of this country and are utilized widely by military services throughout the world. They come in either a tactical missile or ballistic missile configuration. Tactical missiles use propulsion systems which most often employ solid propellants to provide thrust to the missile. Solid propellants are classified into two broad categories; homogenous and heterogenous. Heterogenous propellants are usually called composite propellants and contain a mechanical mixture of inert binder, an oxidizer, and in many cases a metal additive.

Metals are added to increase the specific impulse and to suppress combustion pressure oscillations. However, there is a reduction in the specific impulse efficiency. Most of this decrease can be attributed to the thermal and velocity lags of the particulate products as they pass through the nozzle and to incomplete combustion of the metal in the combustion chamber [Ref. 1]. Two metal additives used are zirconium carbide and aluminum.

Zirconium carbide is often used when there is a need for a reduced signature propellant. But it is not known exactly how ZrC acts in the motor environment. One possibility is that as propellant is burned, oxygen from the ammonium perchlorate (AP) oxidizer flame combines with the carbon to produce carbon monoxide (CO) or carbon dioxide (CO_2). The oxygen then reacts with zirconium to produce a porous ZrO_2 surface layer. Zirconium has a higher boiling point temperature than its oxide, so surface combustion could be expected to occur with an incandescent particle. [Ref. 2]

Aluminum is the most commonly used of all metal additives. As the propellant burns the oxygen from the ammonium perchlorate reacts with the aluminum to form Al_2O_3. A droplet may be formed with a molten aluminum core and a Al_2O_3 liquid outer shell. Aluminum has a lower boiling point temperature than its oxide, which results in a gaseous diffusion flame around the particle.

The burning propellant can also produce agglomerates which can contain thousands or even millions of the original particles. The size of the agglomerates is a function of many factors such as propellant type, original particle size, chamber pressure, burning rate, and metal additive concentration. These agglomerates can later break up in the converging section of the nozzle. [Ref. 3]

This investigation was part of a study to determine the behavior of particulate material in solid propellant rocket motors. The propellants contained either 1.00% zirconium carbide, 2.00% aluminum or 4.68% aluminum. The zirconium carbide propellant was provided by the Naval Weapons Center, China Lake, California. The aluminized propellants were provided by the Air Force Astronautics Laboratory (AFAL), Edwards, California.

Many diagnostic techniques and propellant configurations have been used to study solid propellant combustion. No one method by itself can be used to determine all of the effects of the metal in propellants. A combination of (1) combustion bomb tests of propellant strands using high speed motion pictures, (2) collection of particulate leaving the burning surface of propellant strands and their subsequent study using light diffraction techniques and a scanning electron microscope (SEM), and (3) a two-dimensional rocket motor with high speed motion pictures was used in the present investigation.

The high speed motion pictures taken of propellant strands in the combustion bomb were used to obtain the flame envelope sizes, particle speeds close to the propellent surface, to determine a burning rate and to observe the formation and ignition of metallic agglomerates. Combustion residue was collected in the combustion bomb by firing the propellants into a device that quenched the particles to prevent further reaction. A Malvern particle sizing system was then used to try to determine the particle size distributions of the residue samples suspended in liquid. The residues were also examined using a SEM for particle size distributions.

A series of high speed films were taken in the converging section of a two-dimensional rocket motor nozzle. The films were then used in an attempt to determine the location of the primary breakup of the metallic agglomerates.

II. EXPERIMENTAL APPARATUS

The combustion bomb used for this investigation was constructed of two pieces of stainless steel with a core volume of 90 cubic inches and an inside diameter of 3.5 inches. The bomb had two large windows (2.4 inches in viewing diameter) and three small windows (1.5 inches in viewing diameter). O-ring seals were used with each of the windows and at the junction of the two pieces of the combustion bomb. Figure 1 is a schematic of the combustion bomb [Ref. 4:p. 66]. Figure 2 is a schematic of the combustion bomb window angular relationship [Ref. 4:p. 67]. Figure 3 is a photograph of the combustion bomb. The maximum operating pressure for the combustion bomb was 800 psi and the maximum pressure used in this investigation was 750 psi.

The combustion bomb pressure was controlled using a dome loaded regulator located in the control booth. A gage showing the bomb pressure was also located in the control booth. Figure 4 contains a picture of the control booth. An actuator valve was pressurized to 100 psi and set to engage from the control booth to introduce a nitrogen flow into the bottom of the combustion bomb through a porous plate. The nitrogen flow was then exhausted through the top of the combustion bomb, through an exhaust line, and then to the outside atmosphere.

This exhaust or purge flow rate was regulated manually by a valve located in the exhaust line.

The propellant strand ignition system consisted of two copper electrodes, with the propellant strand mounted between the electrodes. A 0.008 inch diameter nickel-chromium ignition wire was then strung between the electrodes to make contact with the top of the propellant strand. Figure 5 contains a photograph of the propellant strand ignition setup. A gun powder/glue mixture was used to insure contact between the ignition wire and the propellant strand and to ensure rapid and uniform ignition of the propellant surface. The ignition wire was connected in series with a variable resistor to control the current provided by a 12 volt DC wet cell battery. A continuity check could be made from the control booth to verify a complete circuit existed. [Ref. 5]

A Hycam model K2004E-115 manufactured by Red Lakes Corporation, Morgan Hill, California was used for the high speed motion pictures. A framing rate setting of 10,000 frames per second was used with 100 foot rolls of Eastman Ektachrome Video News Film, High Speed 7250 Tungsten by Kodak. The camera gave a framing rate that accelerated to maximum. It achieved a framing rate of approximately 6,000 frames per second by the end of a 100 foot roll. A setting of 11,000 frames per second was used with 400 foot rolls of film, which achieved a framing rate of 7,000 frames per second during the recorded event. A Red Lakes Millimite TLG-4 oscillator was connected to the camera to provide timing marks on the edge of the film at a rate of 1,000 per second. A Nikon Zoom-Nikor 35-105mm lens, mounted inverted, was used with the Hycam. The lens could be adjusted to touch the viewing window if desired. The best focal distance was found to be 5.2 inches from the propellant strand. This gave a magnification of 2.0 and a depth of field of +/- 3.7 millimeters.

Lighting was provided by a Selectroslide model SLM-1200 1200 watt lamp manufactured by Spindler and Sauppe, Glendale, California and by a 150 watt arc lamp made by Sage Action Inc., Ithaca, New York with a collimating shroud and a Varian illumination power supply. The SLM-1200 only was used to light the surface of the zirconium carbide propellant. The light was focused through the small window located at 45 degrees from the viewing window used for the filming, providing light to the front surface of the propellant strand. Figure 6 is a photograph of the setup used for the zirconium carbide firings and Figure 7 provides a schematic of the lighting setup used. A combination of the SLM-1200 and the arc lamp was used in an attempt to overpower the flame envelopes of the aluminum propellants. The arc lamp provided illumination through the 45 degree window to the front surface of the propellant strand. To accomplish this a mirror had to be used in conjunction with the arc lamp due to physical limitations of the work area. An infrared filter and a focusing lens were used to obtain the brightest possible lighting and keep the propellant from being heated at the same time. The SLM-1200 was placed to provide illumination parallel to the front surface of the propellant strand. Figure 8 contains a schematic of the lighting setup used for the aluminized firings and Figure 9 is a photograph of the lighting setup.

Ignition of the propellant strand, pressurization of the combustion bomb and operation of the high speed camera were controlled from a panel located in a control booth for safety reasons. The booth provided viewing of the combustion bomb through a one inch thick Plexiglas window. Figure 10 is a photograph of the remote control panel. The remote control panel provided both manual and automatic start options. The current firings were conducted with the camera in the automatic mode, with an adjustable time-delay between the camera initiation and propellant strand ignition. This allowed ignition and burning of the strand to occur during the maximum framing rate of the film. Combustion residue was collected using a stainless steel collection cup designed and built for this bomb. The cup had a one inch diameter and was 1.18 inches in depth. Fluid to a depth of 0.50 inches was placed in the collection cup. Water was used with the zirconium carbide collections and isopropanol alcohol was used with the aluminum collections. The propellant strands were loaded upside down to fire directly into the fluid, with approximately 0.5 inches from the surface of the propellant strand to the surface of the fluid. The combustion bomb was surized with nitrogen and a slight flow of nitrogen was used to keep the alcohol from igniting when it was used with the aluminum as a quenching medium. Figure 11 is a photograph of the residue collection system and Figure 12 is a schematic of the residue collection apparatus. The same propellant strand ignition system was used except no gunpowder/glue was needed for these firings. This also kept contamination of the residue down to a minimum. The collected specimens were ultrasonically cleaned using methanol as a bath.

After the residue was cleaned, the specimens were analyzed using light diffraction techniques (a Malvern 2600 Particle Sizer with version 3.0 software). A 100 millimeter lens was utilized which gave a resolution of particles down to one micron in diameter, and a particle in liquid setting was used. The samples were placed in a distilled water container with a magnetic stirrer keeping the particles in suspension during sampling. Figure 13 contains a schematic of the light diffraction residue sizing setup and a photograph of the setup is shown in Figure 14.

The residue specimens were dried in a Hotpack Model 1262 refrigerator/oven. The dried particles were prepared for electron microscope analysis by depositing them on a thin layer of epoxy and then gold coating with a Denton Vacuum Evaporator (and a DSM-5 sputter module to insure an even coating was obtained). SEM observations were then made using both a Hitachi S-450 and a Stereoscan 200 made by Cambridge

Instruments, Buffalo, New York, but all SEM photographs were made using Polaroid Type 52 film with the Hitachi S-450.

A two-dimensional rocket motor [Ref. 6] was used with the high speed camera in an attempt to determine the primary breakup point of the agglomerates in the converging section of the nozzle. High speed motion pictures were taken through a 0.343 inch viewing window. Nozzles were designed and constructed to place the expected breakup point within the viewing window. A nitrogen purge was used to keep the windows clear during firing. Figure 15 is a photograph of the window and nozzle area. Figure 16 is a schematic of apparatus setup and Figure 17 is a schematic of the two-dimensional rocket motor. Figure 18 provides a photograph of the two-dimensional motor filming setup.

III. EXPERIMENTAL PROCEDURES

A. COMBUSTION BOMB HIGH SPEED MOTION PICTURES

Three propellants were investigated. The propellant compositions are listed at Table I. Three pressures were selected for the study: 100 psi, 500 psi and 750 psi. The propellant strands were sized to the camera viewing field, and a 45 degree cut was made to the forward portion of the top surface of the strand to give a better view of the burning surface. The rest of the top of each propellant strand was cut flat to aid in placement and subsequent ignition of the strand. The strands were then glued with a wood base cement to stainless steel posts. Figure 19 gives the dimensions of the propellant strands [Ref. 5:p. 60]. Two types of posts were used for mounting the propellant strands. One was a circular pedestal and one had the pedestal modified into a semi-circle. The modified pedestal was used to increase purge flow next to the aluminum propellant strand surface. The propellant strand ignition system was set up as detailed in the Experimental Apparatus section.

Propellants strands were burned in a nitrogen environment in the combustion bomb. High speed motion pictures for the zirconium carbide propellant were taken at each pressure under the lighting conditions described in the Experimental

Apparatus section. A medium flow of nitrogen purge was used to keep the viewing windows clear during the filming. High speed motion pictures for the two aluminum propellants were taken at each pressure using both types of mounting pedestals, giving two films for each pressure setting. A high nitrogen purge was necessary for the aluminum propellant strand films.

The high speed camera was set at a framing rate of 10,000 frames per second for the combustion bomb films. The eyepiece of the camera was focused on the cross hairs of the accessory focusing screen prior to the combustion bomb tests. All tests were conducted with the camera viewing 90 degrees from the propellant strand front surface. An aperture setting of f-5.6 was used with the zirconium carbide propellants and the SLM-1200 was used to provide illumination to the front surface of the propellant strand. An aperture setting of f-16 was used with the aluminum propellants and a 150 watt arc lamp was used for front surface illumination with the SLM-1200 used to provide side illumination. The Millimite timer was set at 1,000 cycles per second for all tests. The best resolution of the propellant strand surface was found to be at 5.2 inches from the camera lens to the front of the propellant strand. Final focusing was done with the camera lens before each firing and just prior to loading film.

The actual ignition and operation of the high speed camera was conducted from the control booth for safety reasons. A timing circuit delayed ignition of the propellants by approximately 0.2 seconds after camera start to more effectively utilize the high framing rates at the end of the film. This was particularly important with the aluminum propellants due to the higher burning rates of these propellant strands.

The films from both the combustion bomb and the two-dimensional rocket motor test were mailed to the Naval Weapons Center, China Lake, California for film processing. Table II shows the test conditions used for the high speed motion pictures. Additional tests are shown at Table III for information in future use of this camera/lens arrangement.

B. RESIDUE COLLECTION

Particle collection was conducted separately from the high speed motion pictures due to the collection cup configuration shielding the viewing surface, and the low purge rate. The combustion bomb and the collection cup were cleaned prior to each firing with acetone. The collection cup was placed in the combustion bomb as shown in Figure 11 and Figure 12 and the ignition wire was positioned in contact with the propellant strand. A propellant strand 7 millimeters in width, 10 millimeters in height, and 5 millimeters in thickness was used for all particle collections. No gun powder glue was necessary to aid in ignition since timing of ignition was not as critical as it was for the high speed motion pictures. A fluid was funneled into the bottom of the collection cup to a depth of 0.5 inches. Distilled water was used for the zirconium carbide propellant residue collections and isopropanol alcohol was used as a quenching medium for the aluminum propellant residue collections. The combustion bomb was pressurized with nitrogen with a purge turned on slightly to insure that all oxygen was removed, then the purge was shut off during propellant strand burnings. Collections were made at 100 psi, 500 psi and 750 psi for each of the three propellants.

After each firing was completed, the residue was washed with methanol into a glass beaker and allowed to settle for eight hours. Methanol was then siphoned off until only a small amount was left covering the residue. New methanol was added to the beaker and it was placed in an ultrasonic cleaner for 30 minutes. The sample was then allowed to stand for eight hours again, and the process was repeated until the methanol was completely clear after an eight hour settling time.

C. MALVERN RESIDUE PARTICLE SIZING

A container of glass and Plexiglas was made with a 33.8 millimeter fluid width to hold the residue for sizing by the Malvern. Distilled water was used as the fluid for suspension of the residue particles during sampling. A magnetic stirrer was used to keep the heavy residue particles from settling to the bottom of the container. A new background was taken with the distilled water being stirred in a clean container prior to each residue sample being sized. An eyedropper was used to add the residue to the distilled water in increments to insure that data was available for obscuration below 50%, and to avoid saturation of the diodes. This provided mean particle diameter and particle size frequency for each of the propellants at the three test pressures. Malvern data was provided by the Naval Weapons Center for the zirconium carbide in its powder form. The Air Force Astronautics Laboratory provided a sample of the aluminum powder used in the aluminized propellants, and the Malvern was used to obtain a size distribution.

D. SEM SAMPLE PREPARATION AND MICROSCOPY

After the Malvern sizing data were obtained, the residue was washed again for SEM sample preparation and allowed to settle for eight hours. The alcohol and distilled water mixture was siphoned off so that approximately five millimeters of fluid remained. This mixture was then heated in the oven at 96 °C until the residue samples were completely dry.

The SEM samples were mounted on pedestals that were sanded with 240 grit, followed by 360 grit, followed by 400 grit and finished using 600 grit sandpaper. Then the pedestals were polished with a 0.05 micron Al_2O_3 slurry on a rotating cloth to provide a smooth surface for residue sample mounting. Three different mounting techniques were tried. A carbon glue was tried, but discarded due to the extremely short time available to mount the residue prior to the glue drying. A conducting tape was tried, but led to overcharging problems with the SEM when trying to view the residue samples. A clear epoxy that dried in five minutes was chosen for best results. A thin layer of epoxy was spread on the mounting surface with a razor blade at a 45 degree angle. The residue was tapped from the glass beakers onto a clean piece of typing paper and a glass rod was rolled lightly over the residue to break up residue that had adhered together. The residue was then tapped gently onto the epoxy surface and left to dry for at least one hour. The residue mounted on the pedestals were then gold plated for 30 seconds in the DSM-5 sputter module to obtain a thin even coating of gold. This was necessary to provide the good conduction necessary for the SEM.

The residue samples were examined with the SEM at an accelerator voltage of 15 kV and a series of photographs were made using type 52 Polaroid film. A 90 degree incidence beam was used for all photographs and a scale factor was automatically labeled in microns by the SEM.

E. TWO-DIMENSIONAL MOTOR HIGH SPEED MOTION PICTURES

High speed motion pictures of the converging section of a two-dimensional rocket motor nozzle were taken in an attempt to determine the primary breakup point of agglomerates in the nozzle. A new nozzle was constructed to bring the nozzle throat into the viewing window.

The camera was set at apertures of f-11 and f-8 for the aluminized propellants. Apertures of f-5.6 and f-3.5 were used for the zirconium carbide propellants. Four hundred foot rolls of film were used with a filming rate of 11,000 frames per second. This allowed a filming of approximately three seconds of propellant burn time. A nitrogen purge of 0.020 lbm/sec was used to keep the window area clear during the firing.

The two-dimensional motor was loaded with four-inch long slabs of 2.00% aluminum propellant for the camera aperture of f-11. Three-inch long slabs of the 2.00% aluminum propellant were also used with a camera aperture setting of f-8. Three-inch long slabs and both aperture settings were used with the zirconium carbide firings. All propellant slabs were approximately 0.25 inches in thickness and 0.75 inches in height.

IV. RESULTS AND DISCUSSION

A. COMBUSTION BOMB HIGH SPEED MOTION PICTURES

The zirconium carbide propellant did not present major problems in obtaining films though several films were taken to determine the best filming conditions as indicated in Table II and Table III. Smoke was not a problem at any of the three pressures used for the nitrogen purge rate available. The only major problem, once the proper aperture and lighting conditions were determined, was with obtaining good films at 750 psi. It was impossible to determine exactly what was occurring on the burning surface but it appeared that there was a great deal of melting and agglomeration of the zirconium carbide on the propellant surface prior to the particles igniting and lifting off the surface. Figure 20 provides some photographs taken from the zirconium carbide burning at 500 psi.

The 2.00% aluminum propellant and the 4.68% propellant presented several problems in adjustment of the aperture and lighting conditions to overpower the flame envelopes as much as possible. Good films were obtained for both 2.00% aluminum and 4.68% aluminum at 100 psi. A smoke problem was encountered at both 500 and 750 psi. The purge rate available could not keep up with the smoke produced by these propellants. Consequently, only the first few moments of the event were clear for data collection and observation. There appeared to be significantly more near-surface burning at the higher pressures, which added to the smoke problem. It was evident that the aluminum was spherical prior to igniting and lifting off the surface. The flame envelopes showed the classic tear drop configuration associated with a diffusion flame for all pressures. Figure 21 shows photographs of flame envelopes and the burning surface for 2.00% aluminum. Figure 22 shows photographs of flame envelopes and the burning surface of 4.68% aluminum.

High speed motion picture films were used in an attempt to determine burning rates, but the results were inconsistent due to local burning down the vertical surfaces of the strands and, at the higher pressures, excessive smoke. For example the ZrC propellant burning rates were measured to be much less than the know burning rates (0.167 in/sec versus 0.279 in/sec). This apparently resulted from burning down the backside of the strand. The aluminized propellants also did not provide any consistent data. These propellants were known to have a burning rate expression of $r = .08\ P_c^{0.312}$. Thus, pressure-time traces from the closed bomb are much more reliable than the motion pictures for obtaining burning rates. Velocities were also measured for particles near the surface, after the flame envelopes had been fully developed. Each propellant showed an inconsistent trend,with velocities at 500 psi being the highest and at 100 psi being the lowest. The velocities fell in a range of 2-12 in/sec. Table IV provides the observed velocities.

The propellant flame envelope sizes were measured for each of the three propellants. There was a limited number of flame envelopes unobscured for the 500 psi and 750 psi films of the aluminum propellants. Only flame envelopes that were in good focus were counted. The flame envelopes measured were reduced to histograms in Figure 23. However, the flame envelopes obscured by smoke showed approximately the same sizes. The trend for all three propellants was a decreased flame envelope size as the pressure increased, indicating either smaller particles developed at higher pressures or flame envelopes closer to the particle surface. Table V contains the measured flame envelope sizes in terms of D_{32} and D_{43}.

B. RESIDUE COLLECTION

Initially, an open collection device was built and samples were collected. Later this collection device was redesigned into what appears in Figures 11 and 12. The fluid quench collection device proved to be easier to use than the open device, with an increase in the amount of residue collected during the firing. Ignition could be accomplished using only an ignition wire. The residue could then be easily prepared for other testing with a minimum of contamination. This significantly lowered the amount of time necessary to clean the residue.

C. SEM RESIDUE RESULTS

Residue was used to prepare SEM samples. Problems were encountered in mounting the samples due to them sticking together. This made it difficult to size the particles in SEM photographs and to locate a large number of data points. Attempts to remedy this met with only limited success. Example photographs are included for each propellant at each pressure in Figures 24a to 24i. Residue particles were measured and reduced to histograms in Figure 25.

Mean particle size data are presented in Table V. The number of particles counted from the films and from the SEM photogrpahs were small, so that only qualitative comparisons could be made. However, when the histograms from the films and SEM were compared it was interesting to note that the shapes of the distributions for a given propellant were similar (i.e., monomodal, bimodal, bimodal with long tails, etc.). Several interesting trends are apparent from the mean particle size data presented in Table V. For the ZrC propellant the flame envelopes were, on the average, four times as large as the actual particles. Thus, the ZrC additive burns, at least in part, with a detached flame. The ratio of flame size to particle size decreased with increasing pressure, indicating that the particle burning occurred closer to the surface with increased pressure. The aluminized propellants (except for an inconsistency for the 2.00% aluminum propellant at 750 psi) showed similar behavior.

However, the aluminum burning occurred much further from the particle surface. The 4.68% aluminum propellant produced smaller particles than the 2.00% aluminum propellant, but the flames were more removed from the particle surface. This could be the result of less available oxygen. Flame to particle size ratios were 13 and 7 for the 4.68% and 2.00% aluminum propellants at 500 psi, respectively.

D. COMPARISON OF ORIGINAL ADDITIVE SIZES TO GATHERED DATA

The Naval Weapons Center, China Lake, California provided Malvern data on the ZrC powder size distribution. D_{32} was five microns. The SEM data indicated that the particles above the surface were, on the average, twice that size.

The AFAL provided a sample of the aluminum powder and it was also analyzed using the Malvern. A D_{32} of 20 microns was measured. Interestingly, the collected residues from just above the burning strands were generally less than 20 microns. This indicates that very little surface agglomeration occurred and that the particles rapidly burned above the propellant surface. In this regard, another interesting behavior was noted from the films. When aluminum particles first began to "glow" on the surface they had diameters between 1.5 and 2.0 times smaller than the flame envelopes surrounding the detached particle. This apparent "surface size" was still significantly larger than the actual collected mean particle size, indicating that the particles were actually igniting on the surface and forming detached flames.

E. MALVERN RESIDUE PARTICLE SIZING

The residue collected was also analyzed using the Malvern. The resultant D_{32} and D_{43} data is provided in Table V. The equipment available (magnetic stirrer) did not keep the larger particles in suspension for sampling. To offset this problem the sampling was conducted immediately after the sample was placed into the container while the larger particle were still suspended in the fluid. The data were biased to smaller particles for any sampling that was not taken immediately after the residue was added to the holder. The log differences were in the range of two to three after the larger particles had settled, which is highly unlikely under laboratory conditions. The Malvern does measure all particles greater than one to two microns, making it particularly attractive for sizing residue. Figures 26a to 26d contain Malvern data and particle size distributions for samples that had an acceptable log difference and obscuration. The most consistent data were obtained for the propellant with 2.00% aluminum. There the D_{32} values (Table V) from the Malvern and SEM analyses were in good agreement.

F. TWO-DIMENSIONAL MOTOR HIGH SPEED MOTION PICTURES

High speed motion pictures were taken under the conditions shown in Table VI in an attempt to determine the location of the primary breakup of the metallic agglomerates. It was felt that there would be sufficient light inside the motor (particles burning) to make outside illumination unnecessary.

This did not prove to be true. At the lowest aperture setting the particles were too dark for filming. Only occasional reflections from the nozzle exhaust were visible. No data was available to determine the location of the primary breakup of the metallic agglomerates.

V. CONCLUSIONS AND RECOMMENDATIONS

The proper camera aperture and lighting conditions were determined for zirconium carbide and aluminum propellants. High speed motion pictures were able to provide good pictures of surface burning characteristics at low pressures. Flame envelope sizes decreased as pressure increased for zirconium carbide and the aluminum propellants. Ignited particle velocities just above the propellant surface fell in the 2 to 12 in/sec range for the propellants, but had no consistent trend. ZrC was also observed to agglomerate and burn on the surface before lifting off, whereas aluminum particles ignited on the surface and were ejected without agglomeration.

SEM analysis of collected residue resulted in size distributions similar in shape, but much smaller than the flame envelope distributions. Comparison of SEM and high speed motion picture data showed that the ZrC burned in part with a detached flame and that the ratio of flame size to particle size decreased as pressure was increased. Thus, at higher pressures the flame envelopes were closer to the particle surfaces. Aluminum behaved in a similar fashion except that the flames were much farther detached from the particle surfaces. Increased aluminum loading produced smaller particles above the propellant surface, but the flames were more distant from the particle surfaces.

The light diffraction particle sizing techniques did not provide consistent data except for the propellant with 2.00% aluminum, where the D_{32} values were in good agreement with the SEM data. Larger particles did not remain in suspension during sampling, providing artificially low D_{32} values. This technique, however, did show that it could be very useful in providing quick and accurate particle size data when the suspension problem is overcome.

High speed motion pictures were not successful in identifying the primary breakup of the metallic agglomerates in a two-dimensional rocket motor. The correct aperture and light setting must be found to provide this information.

Obtaining particle size data from films taken at high pressures will require a solution to the smoke obscuration problems encountered. A higher regulated purge rate may solve the problem, and different propellant geometries may help also with surface burning problems. The same aperture settings should be used as in previous testing. Burning rate information should be obtained from measured pressure-time traces.

The breakup point of the metallic agglomerates within the two-dimensional motor should be obtainable with the use of a 1200 W light in a rear lighting configuration. Aperture setting of f-8 and f-11 should be tried initially. High purge rates must be used with aluminum propellants to keep the windows clear for the camera.

The residue collection technique of quenching the particles in a fluid to collect a sample was very satisfactory. Many of the problems with the inconsistent data probably stemmed from the low sample population. Much bigger propellant strands should be used and multiple firings into the same fluid should be tried to provide a large residue population for both the Malvern and SEM analyses. A new magnetic stirrer that would suspend the larger particles would correct the majority of the inconsistencies in the Malvern data.

SEM sample preparation was unsatisfactory. The particles agglomerated and it was difficult to get a usable sample. A wetting solution may help, as well as increasing the residue sample.

TABLE I

PROPELLANT COMPOSITION

Constituents	2.00% Al	4.68% Al	ZrC
Gap (200-1)	14.67%	14.67%	
Tegdn (AK-17E)	8.49%	8.49%	
Aluminum (C003)	2.00%	4.68%	
AP (200 micron)	47.45%	45.70%	57.00%
AP (25 micron)	25.55%	24.61%	
AP (11 micron)			25.00%
R45M			8.851%
RDX			4.00%
Dioctyl Adipate			2.00%
Zirconium Carbide (5 micron nominal)			1.00%
DDI			1.76%
Others	1.84%	1.84%	0.389%

TABLE II

HIGH SPEED MOTION PICTURE TEST CONDITIONS

Propellant	Pressure	Aperture Setting	External Lighting
ZrC	100 psi	5.6	1200 W - 45 deg
ZrC	500 psi	5.6	1200 W - 45 deg
ZrC	750 psi	5.6	1200 W - 45 deg
2.00% Al	100 psi	16	1200 W - side 150 W Arc - 45 deg
2.00% Al	500 psi	22	1200 W - side 150 W Arc - 45 deg
2.00% Al	750 psi	22	1200 W - side 150 W Arc - 45 deg
4.68% Al	100 psi	16	1200 W - side 150 W Arc - 45 deg
4.68% Al	500 psi	16	1200 W - side 150 W Arc - 45 deg
4.68% Al	750 psi	16	1200 W - side 150 W Arc - 45 deg

TABLE III

ADDITIONAL HIGH SPEED MOTION PICTURE TEST CONDITIONS
(POOR RESULTS)

Propellant	Pressure	Aperture Setting	External Lighting
ZrC	150 psi	3.5	1200 W - side
	750 psi	8	1200 W - side
	500 psi	22	1200 W - 45 deg
	750 psi	22	1200 W - 45 deg
	150 psi	16	1200 W - 45 deg
	750 psi	16	1200 W - side
Aluminum	750 psi	5.6	1200 W - 45 deg
	500 psi	8	1200 W - 45 deg
	100 psi	8	1200 W - 45 deg with 20% filter
	150 psi	11	1200 W - 45 deg
	500 psi	22	150 W Arc - 45 deg with 20% filter
	100 psi	22	1200 W - 45 deg
	500 psi	16	1200 W - 45 deg
	100 psi	22	1200 W - 45 deg 150 W Arc - side
	500 psi	16	1200 W - 45 deg
	500 psi	22	150 W Arc - 45 deg

TABLE IV

NEAR SURFACE PARTICLE VELOCITIES (IN/SEC)

Propellant	100 psi	500 psi	750 psi
ZrC	6.29 in/sec	12.09 in/sec	6.78 in/sec
2.00% AL	2.12 in/sec	11.12 in/sec	3.17 in/sec
4.68% Al	3.00 in/sec	5.94 in/sec	2.16 in/sec

TABLE V

D_{32} AND D_{43} VALUES

Propellant	Data Source	100 psi	500 psi	750 psi
ZrC	Films D_{32} D_{43}	261* 51 μm 55 μm	253* 46 μm 50 μm	290* 33 μm 35 μm
ZrC	SEM D_{32} D_{43}	17* 10.1 μm 12.5 μm	**	233* 9.4 μm 12.6 μm
ZrC	Malvern D_{32} D_{43}	**	25 μm 102 μm	31 μm 114 μm
2.00% Al	Films D_{32} D_{43}	100* 187 μm 201 μm	83* 134 μm 157 μm	75* 112 μm 125 μm
2.00% Al	SEM D_{32} D_{43}	181* 25.8 μm 33.4 μm	195* 19.4 μm 22.7 μm	44* 11.8 μm 13.3 μm
2.00% Al	Malvern D_{32} D_{43}	21 μm 86 μm	**	14 μm 42 μm
4.68% Al	Films D_{32} D_{43}	125* 111 μm 121 μm	75* 118 μm 133 μm	50* 83 μm 95 μm
4.68% Al	SEM D_{32} D_{43}	193* 6.4 μm 8.0 μm	133* 8.9 μm 13.4 μm	174* 12.3 μm 16.8 μm
4.68% Al	Malvern	**	**	**

Note: * Indicates the number of particles, in focus, counted. ** Indicates no consistent data obtained

TABLE VI

TWO-DIMENSIONAL ROCKET MOTOR HIGH SPEED MOTION PICTURE FILMS

Propellant	Slab Length	Aperture Setting	External Lighting
ZrC	3 in	5.6	none
	3 in	3.5	none
2.00% Al	4 in	11	none
	3 in	8	none

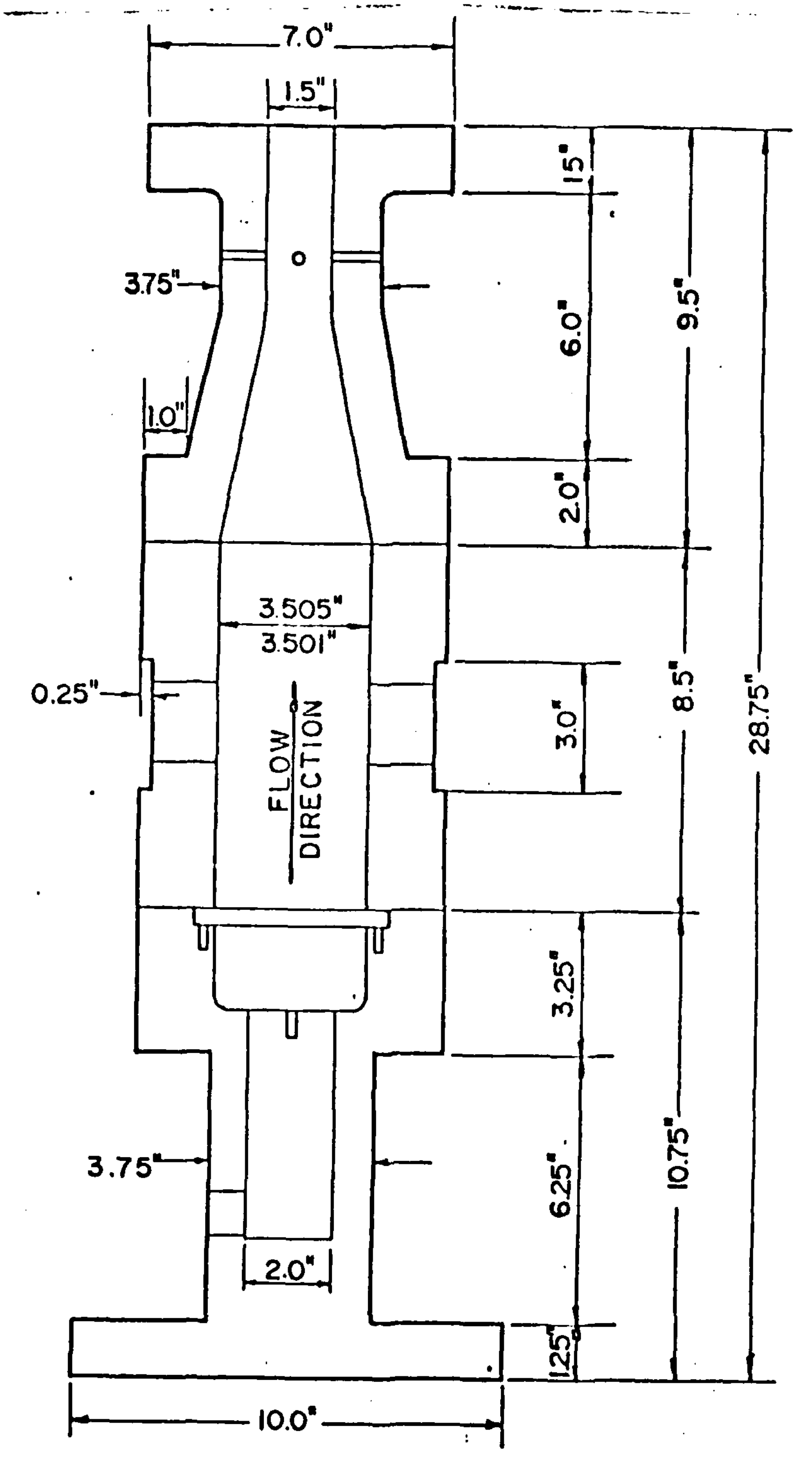


Figure 1. Combustion Bomb Schematic

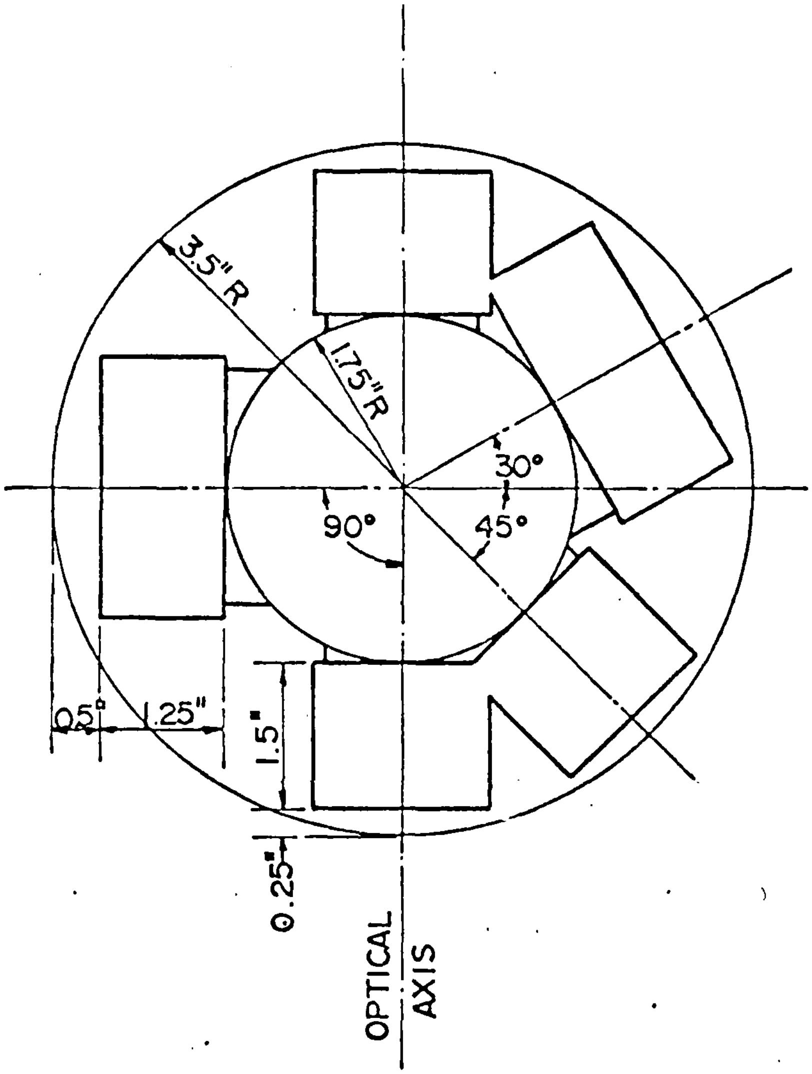


Figure 2. Angular Relationship of Combustion Bomb Windows

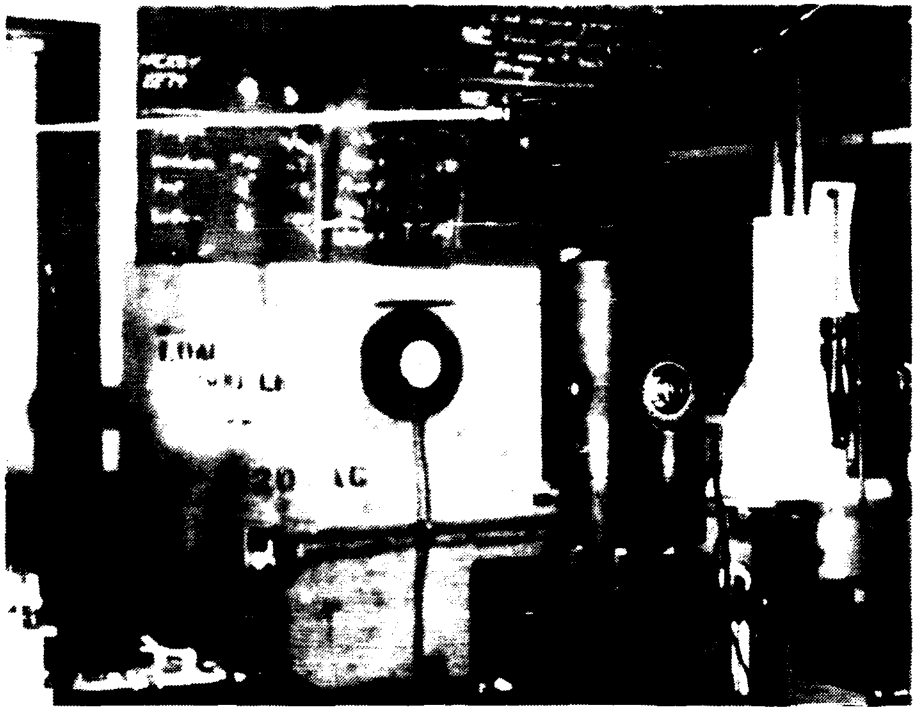


Figure 3. Photograph of Combustion Bomb

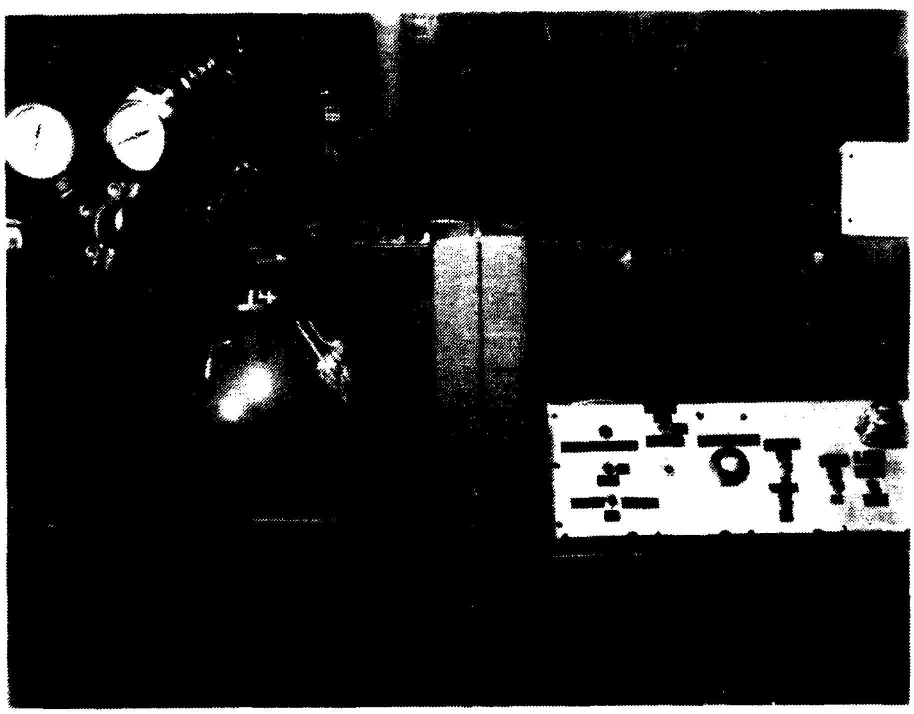


Figure 4. Photograph of Control Booth

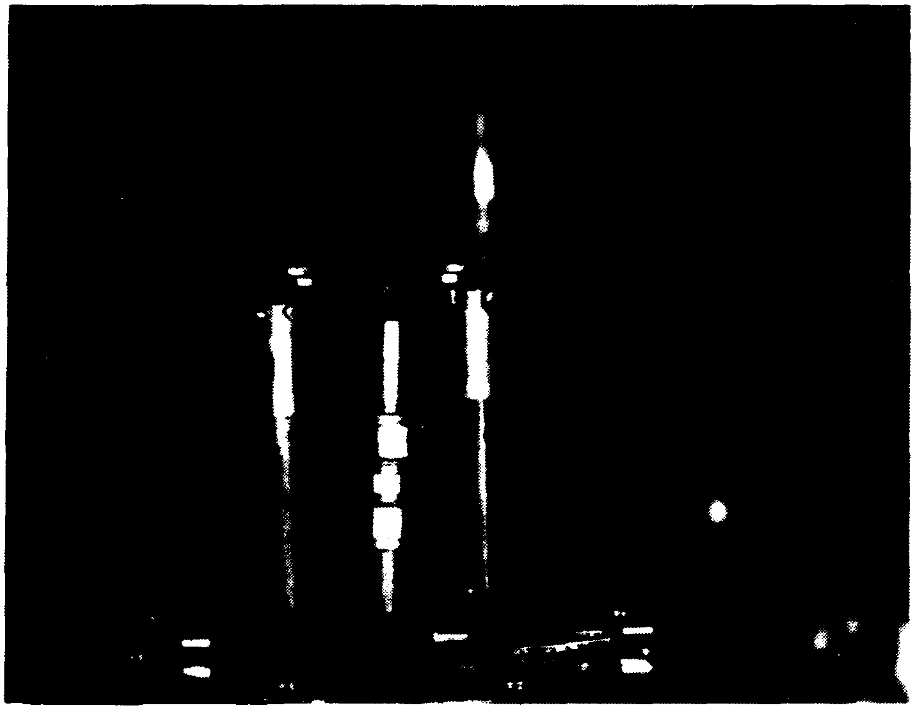


Figure 5. Photograph of Propellant Strand Ignition Setup

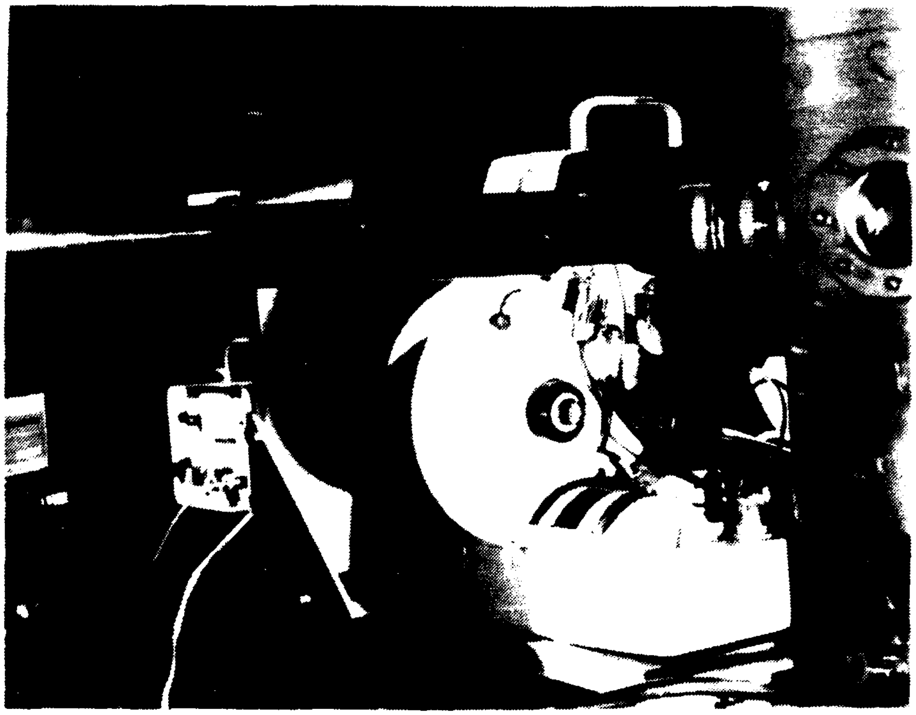


Figure 6. Photograph of ZrC Lighting Setup

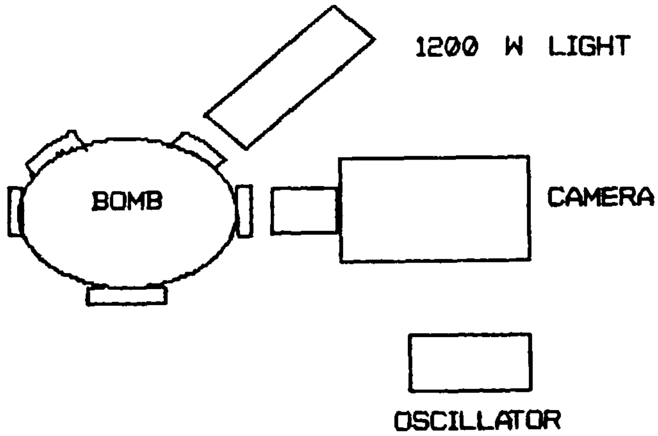


Figure 7. Schematic of ZrC Propellant Lighting Setup

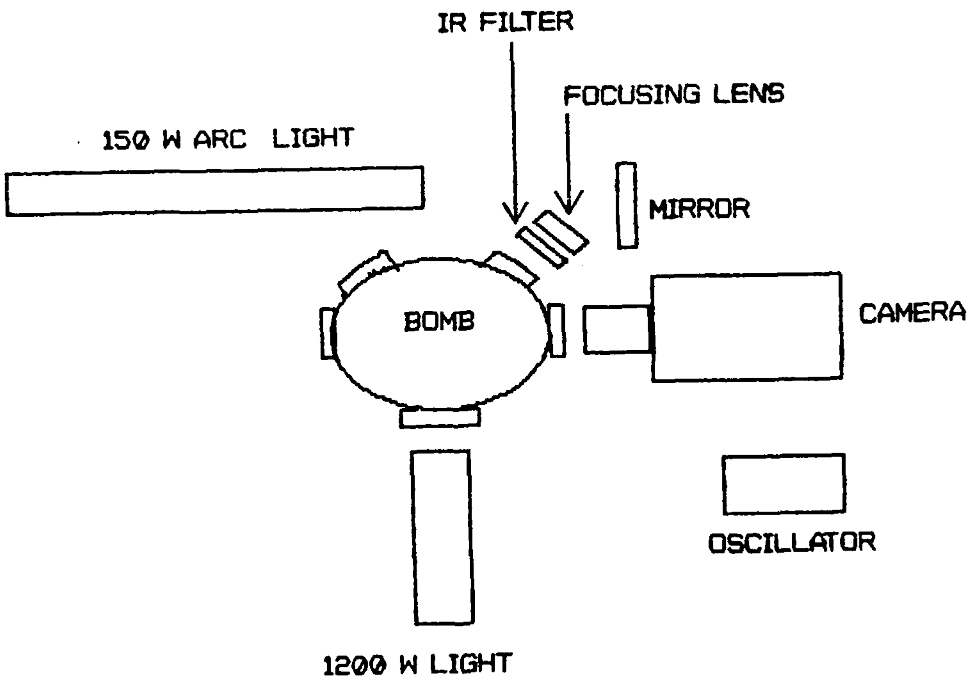


Figure 8. Schematic of Aluminum Propellants Lighting Setup

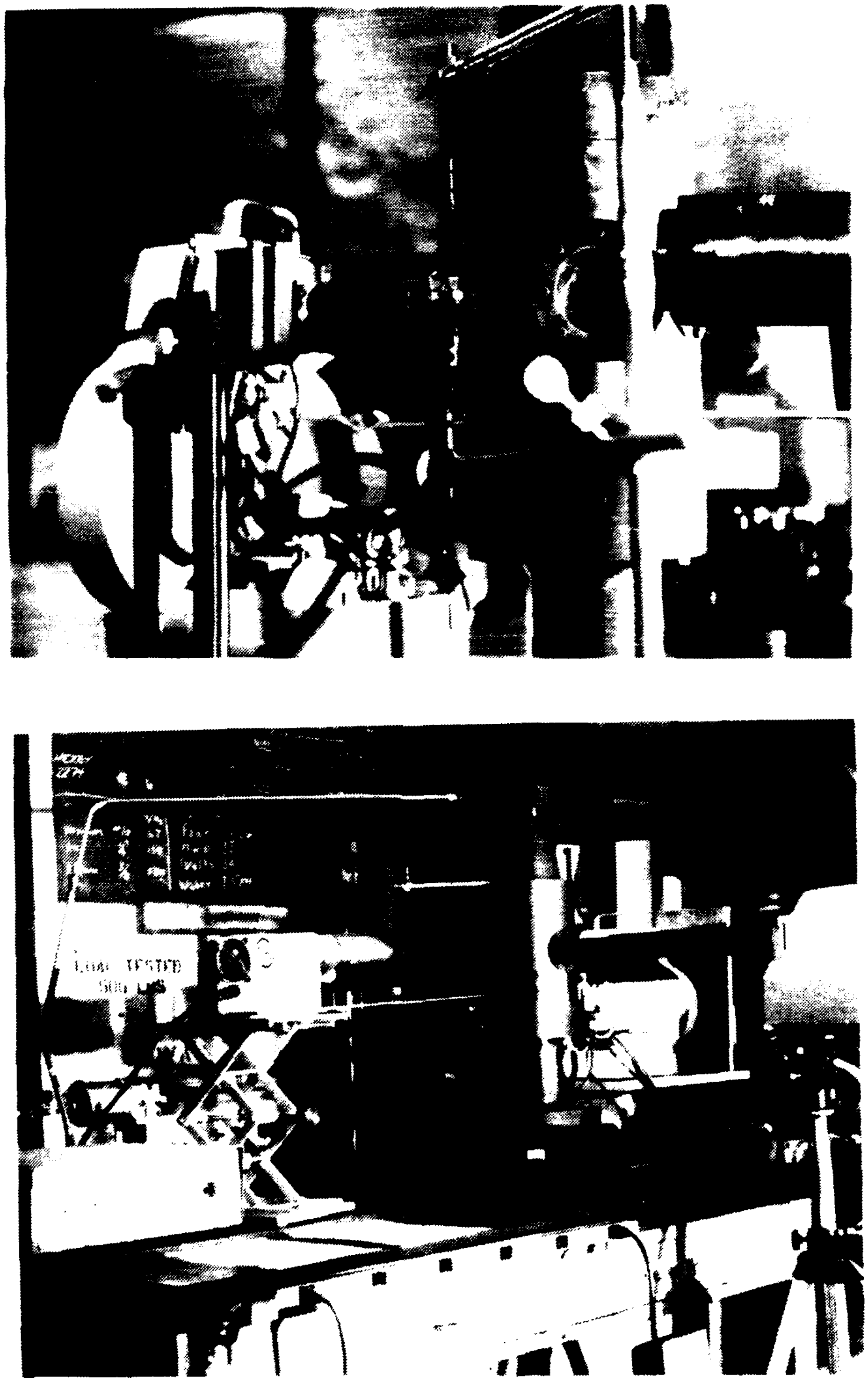


Figure 9. Photographs of 2 Views of Aluminum Lighting Setup

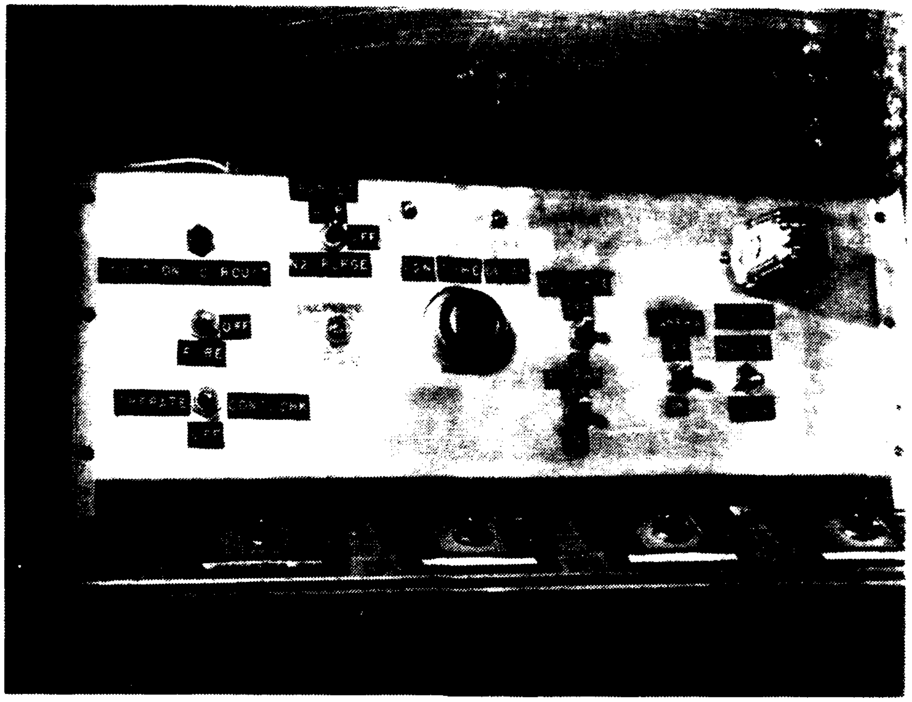


Figure 10. Photograph of Remote Control Panel

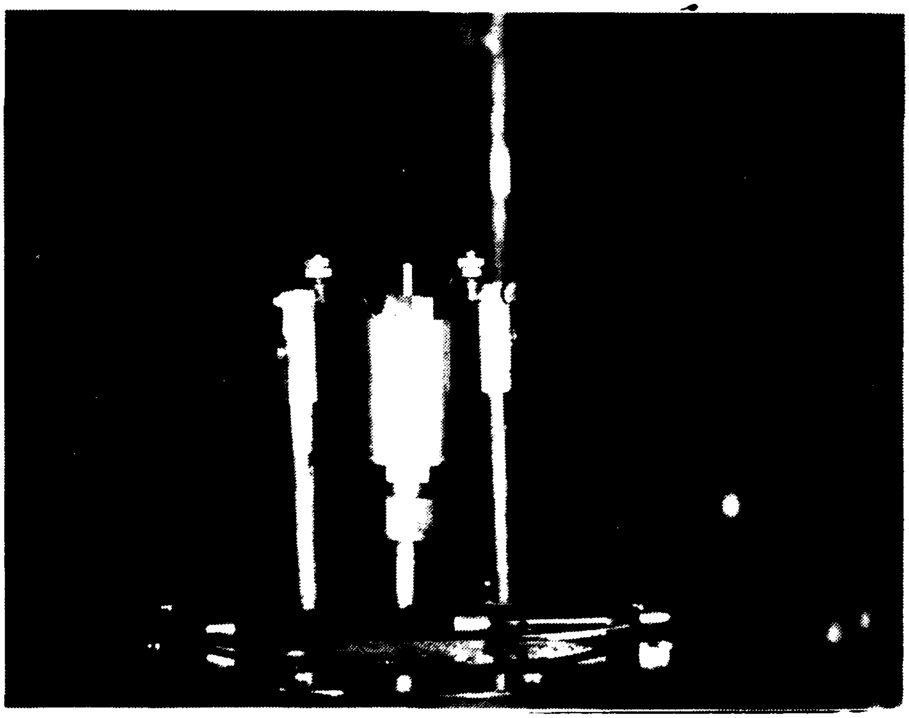


Figure 11. Photograph of Residue Collection Apparatus

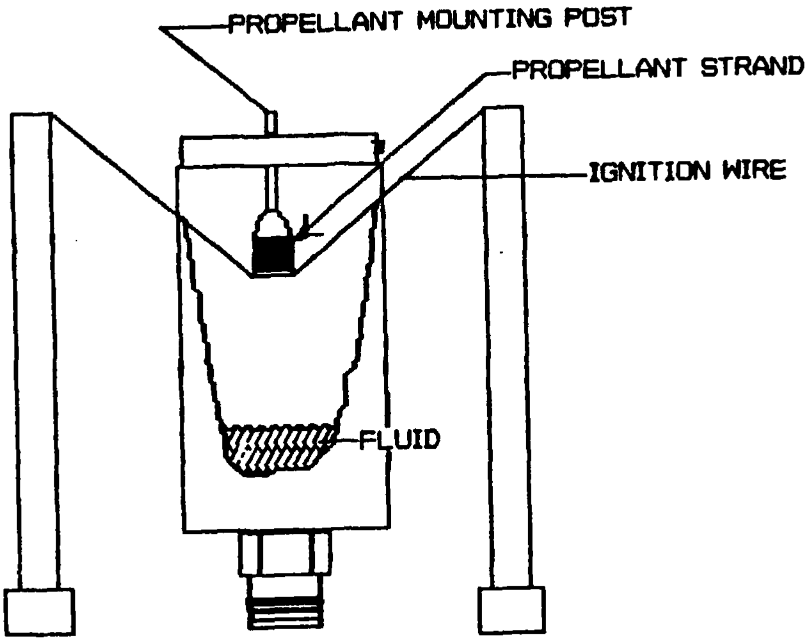


Figure 12. Schematic of Residue Collection Apparatus

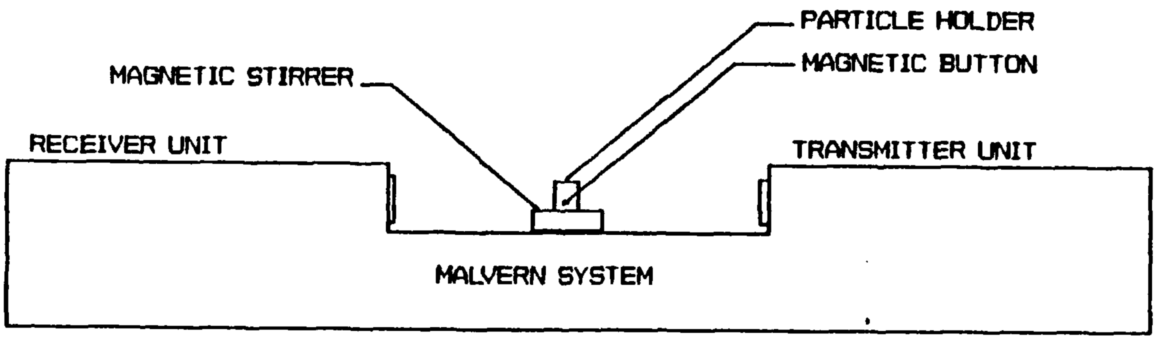


Figure 13. Schematic of Residue Particle Sizing Setup

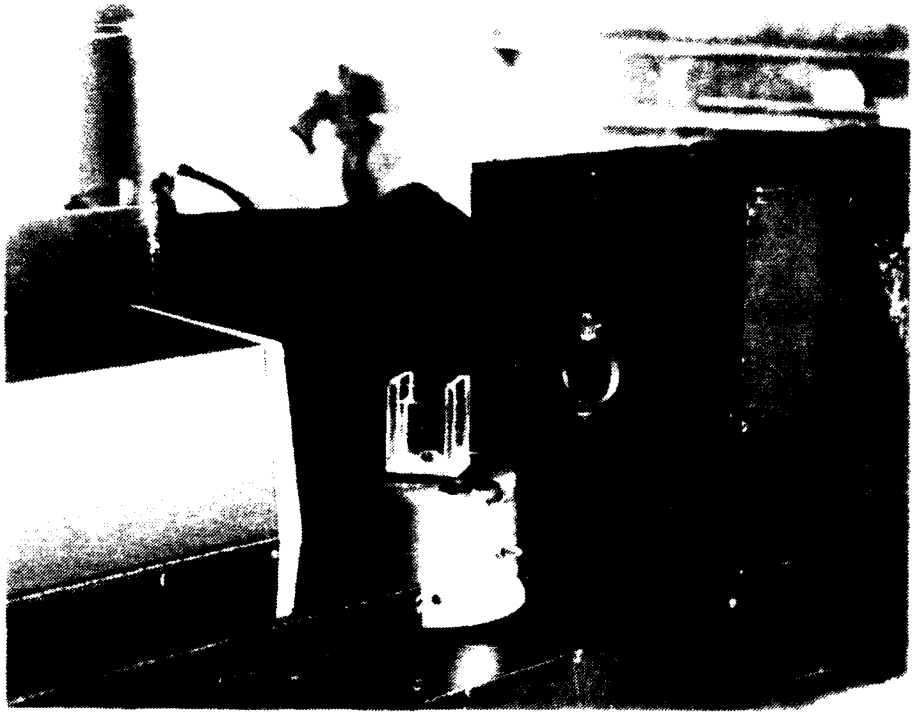


Figure 14. Photograph of Residue Particle Sizing Setup

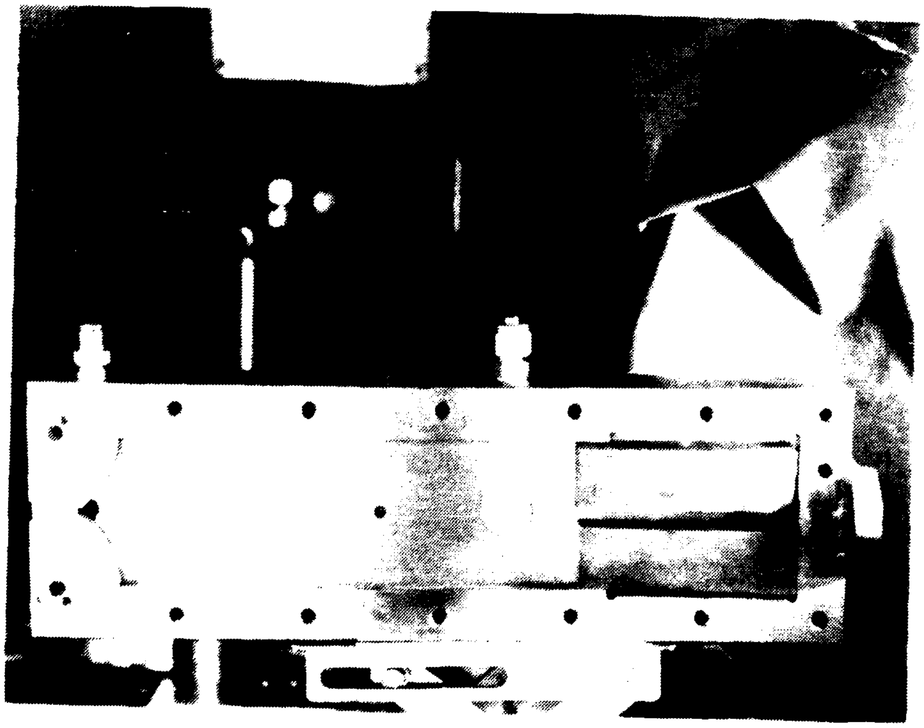


Figure 15. Photograph of Two-Dimensional Rocket Motor Firing Setup

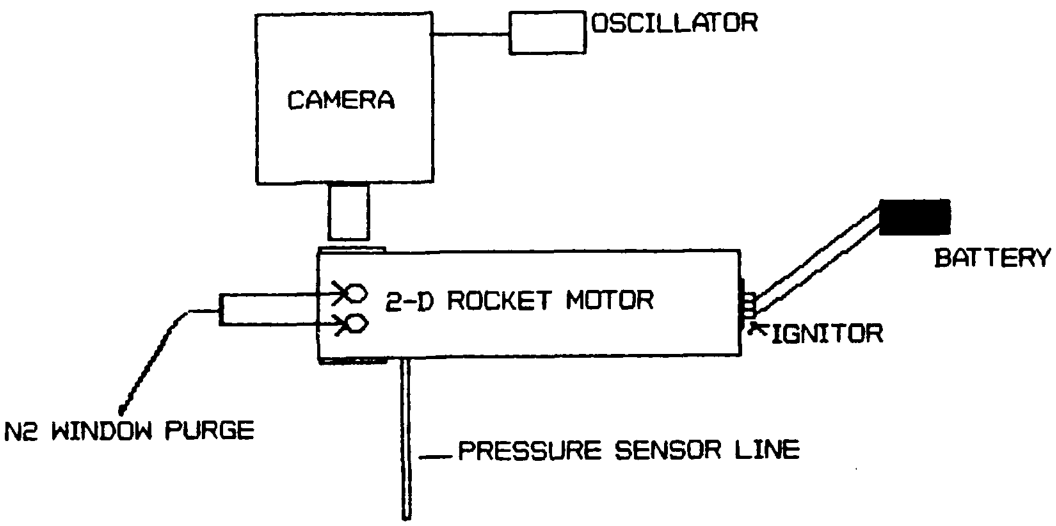


Figure 16. Two-Dimensional Rocket Motor Firing Setup

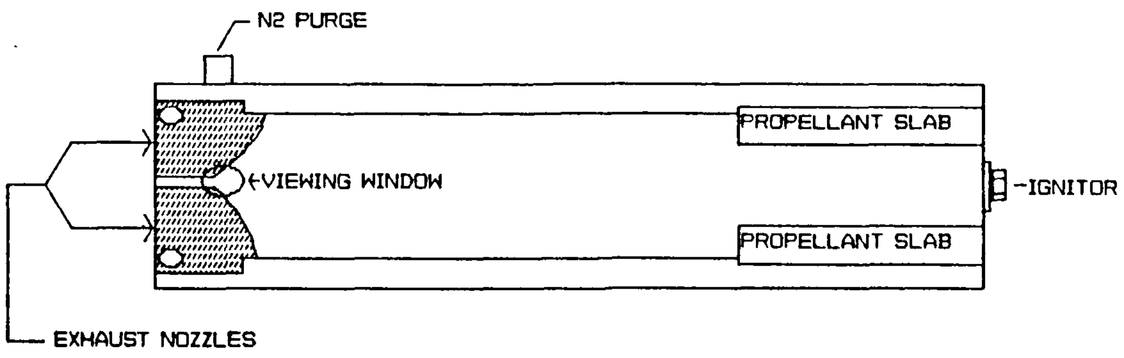


Figure 17. Schematic of Two-Dimensional Motor Interior

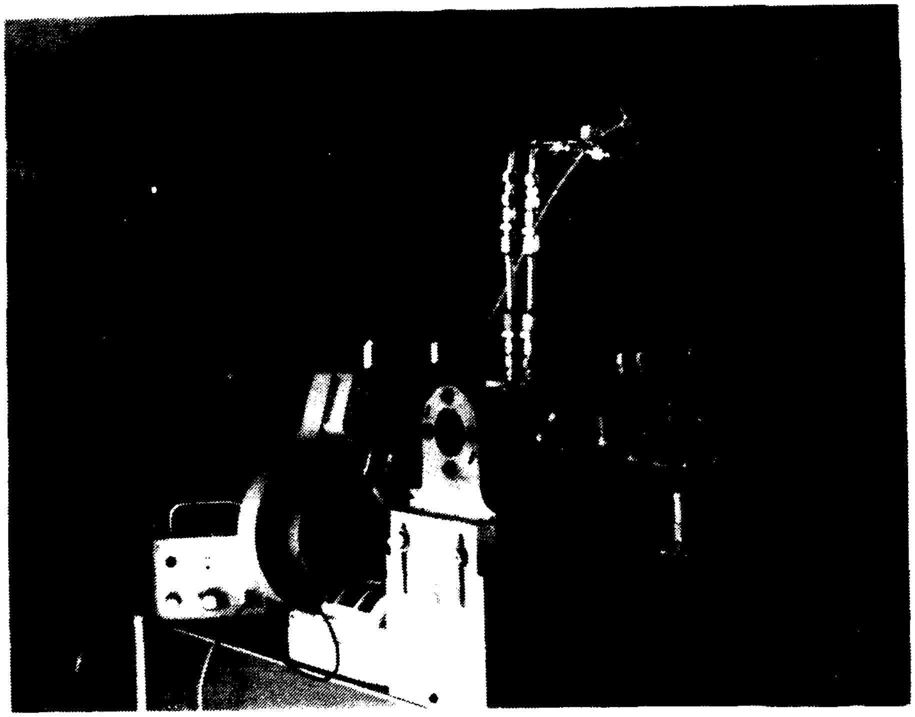


Figure 18. Photograph of Two-Dimensional Rocket Motor Setup

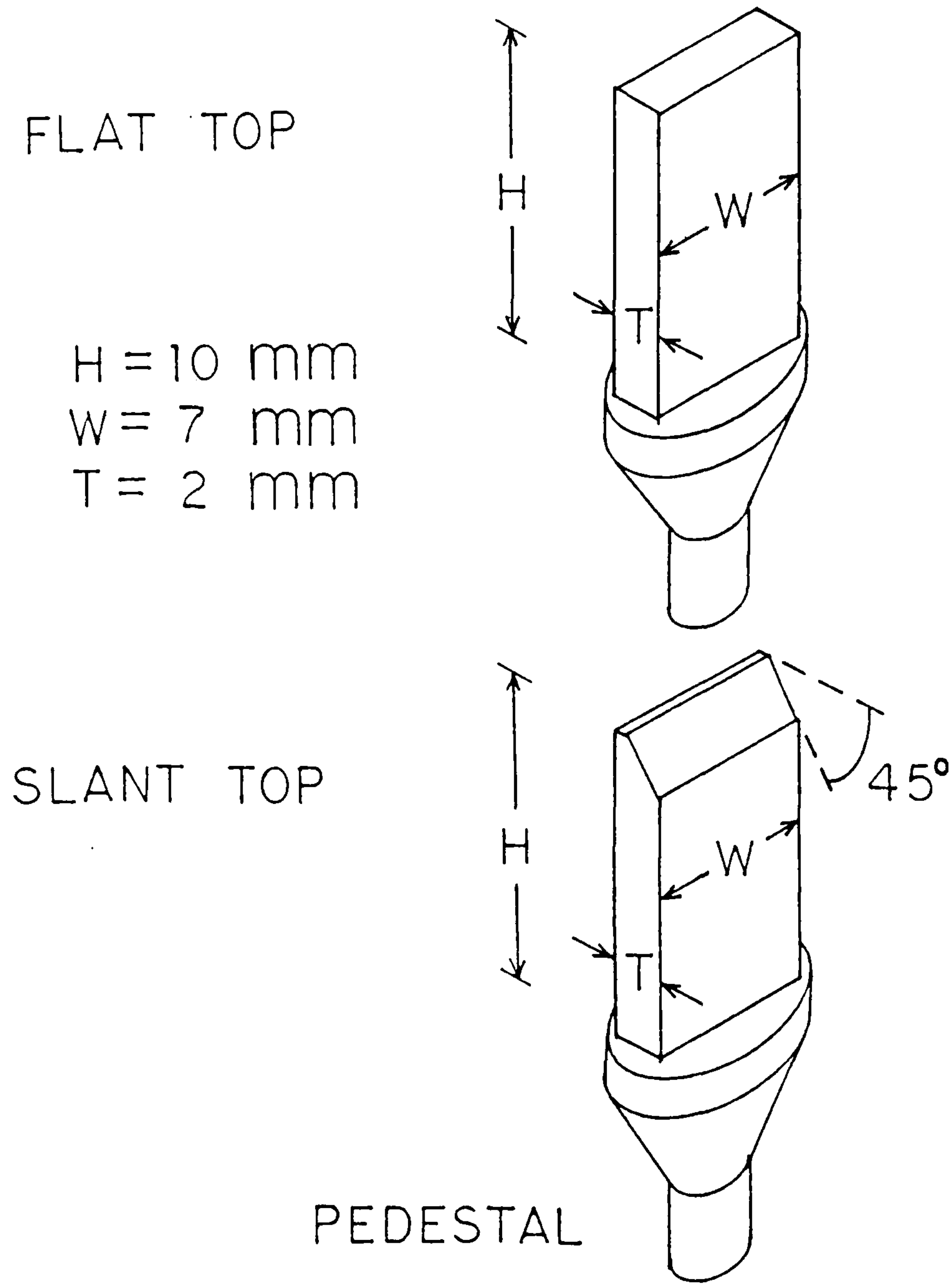


Figure 19. Dimensions of Propellant Strand

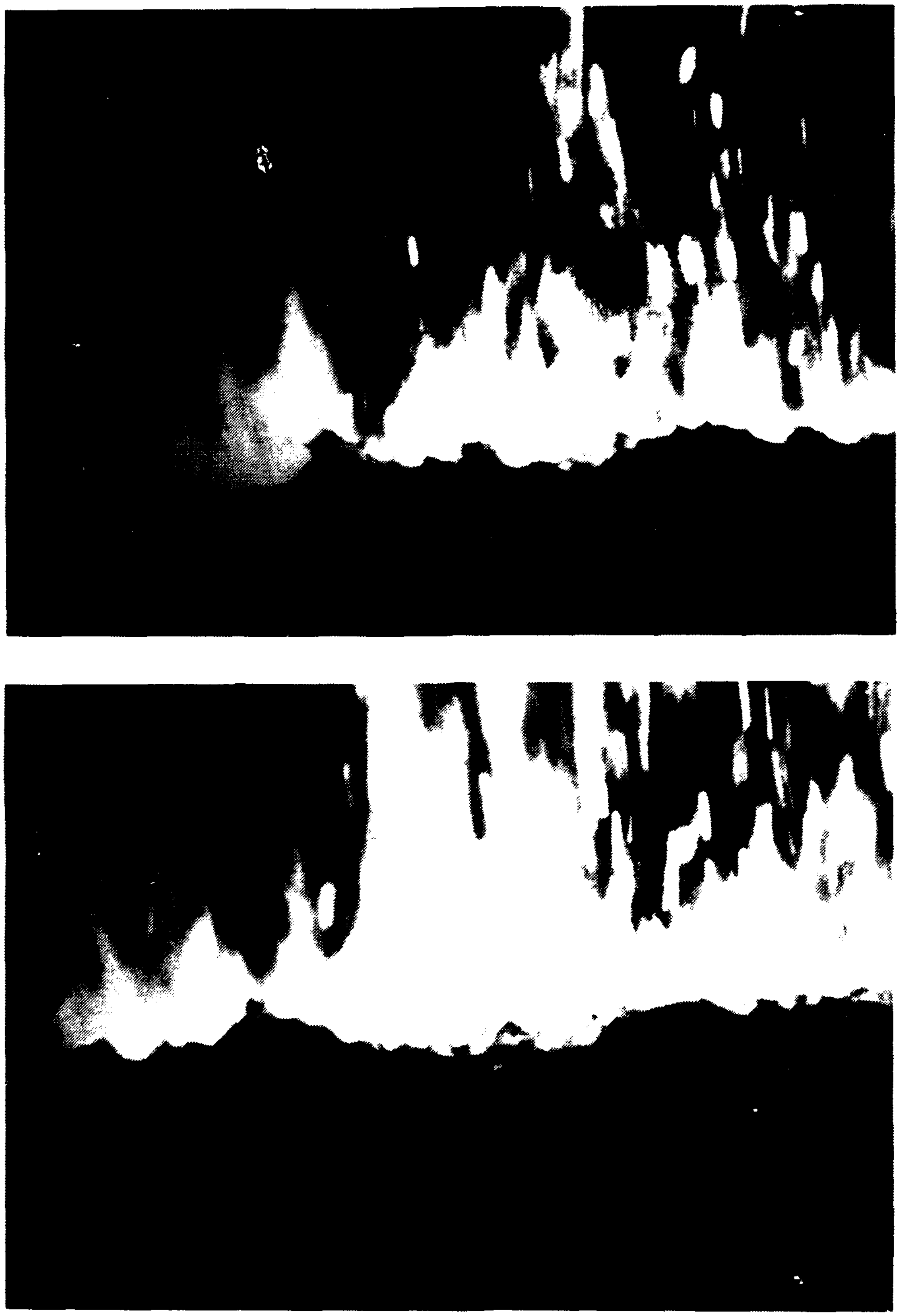


Figure 20. ZrC Flame Envelope Photographs at 500 Psi

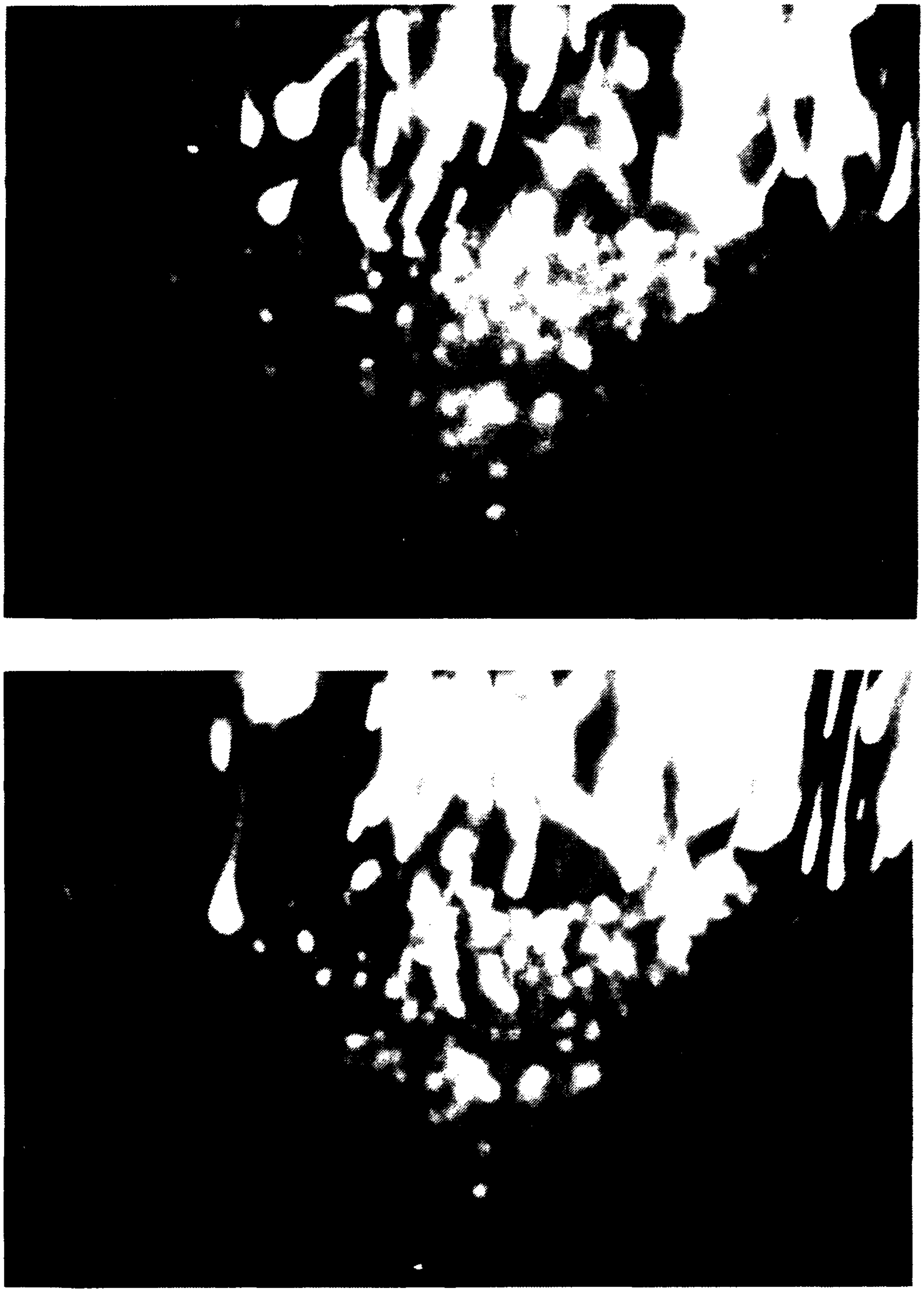


Figure 21. 2.00% Al Flame Envelope Photographs at 100 Psi

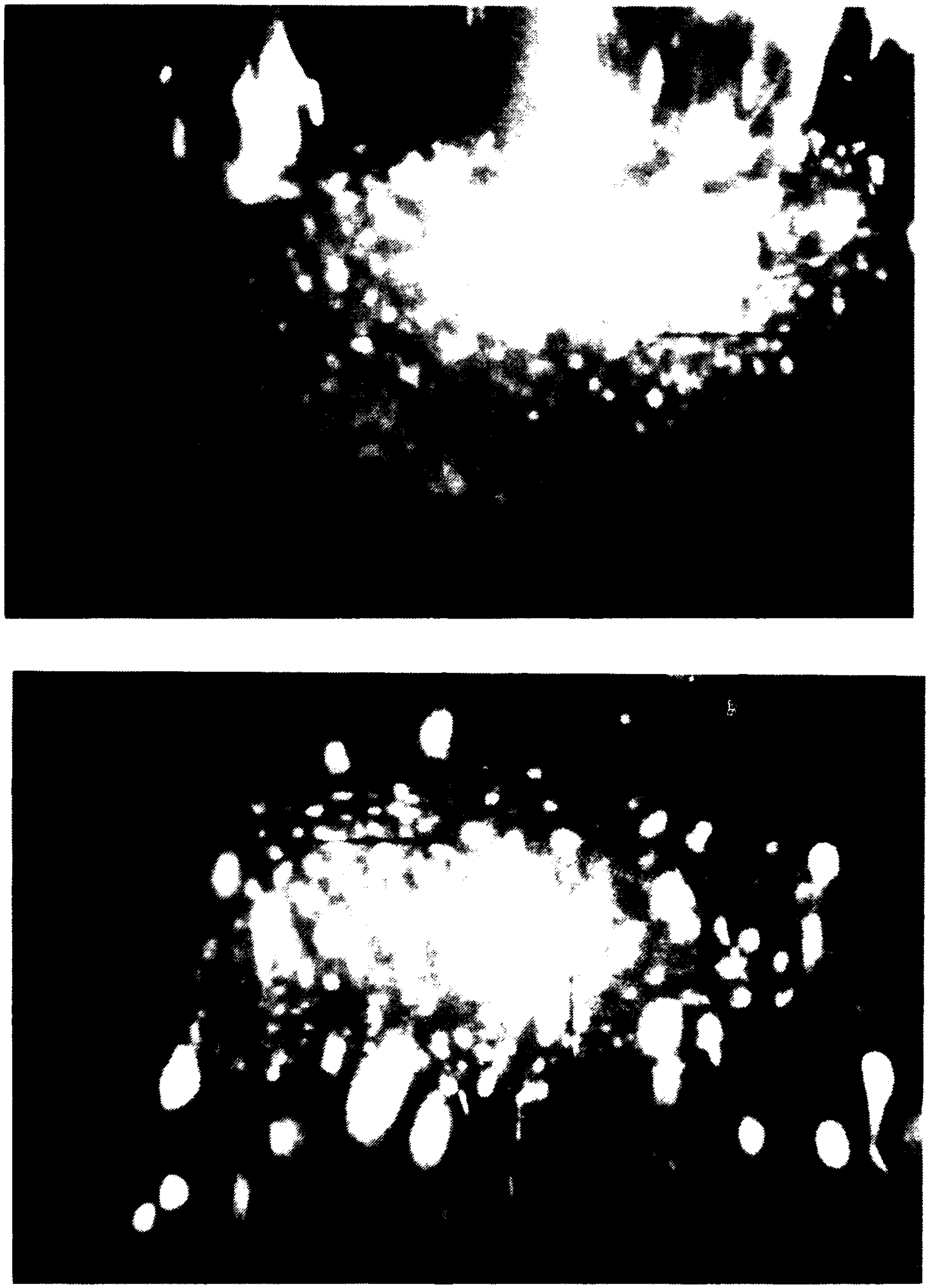
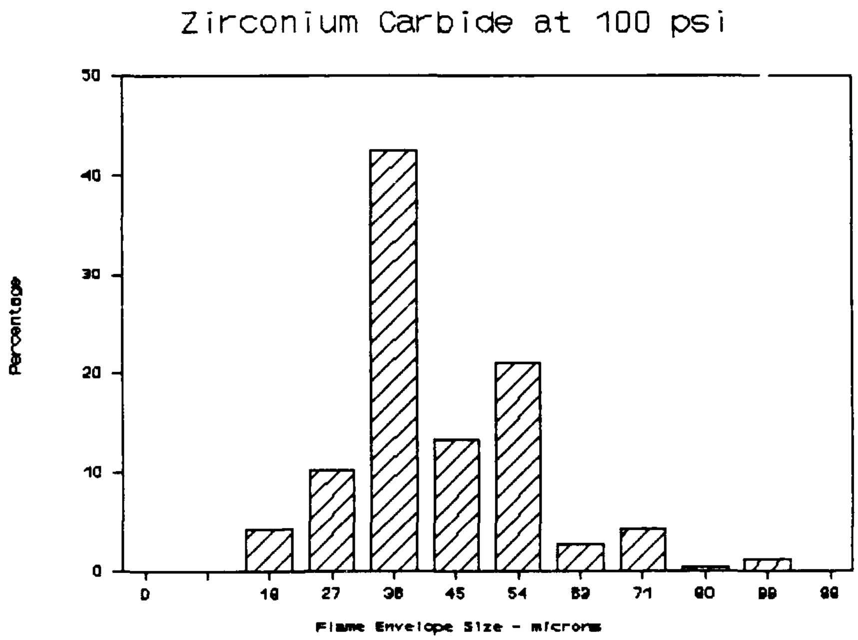


Figure 22. 4.68% Al Flame Envelope Photographs at 100 Psi



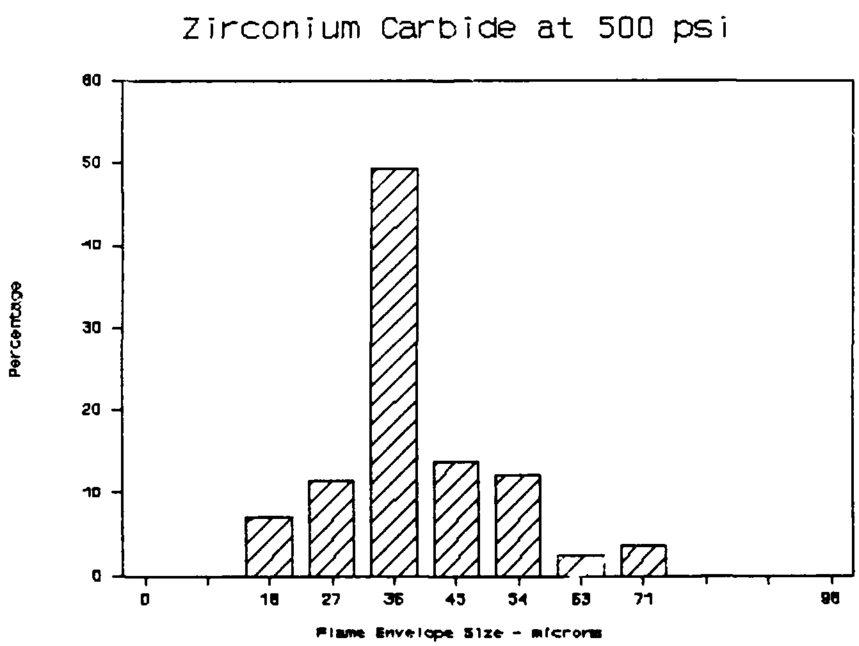
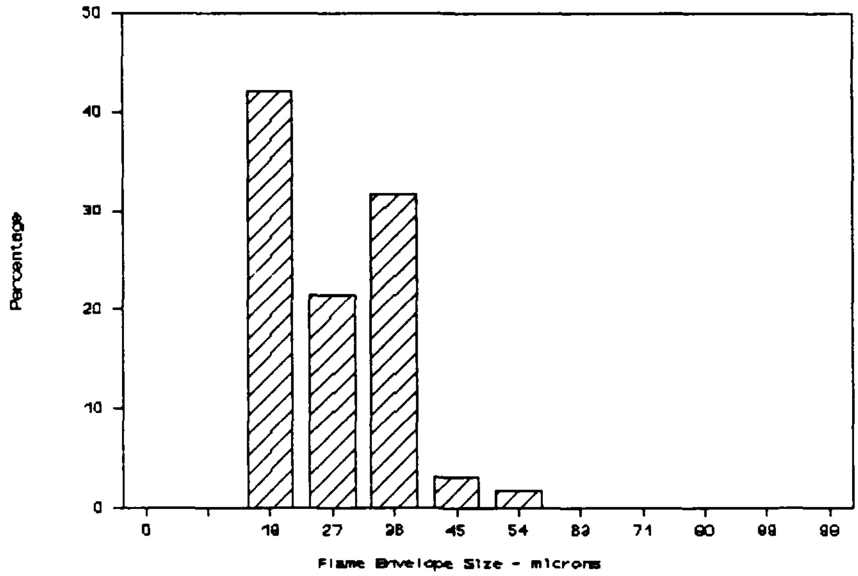


Figure 23. High Speed Film Flame Envelope Sizes

Zirconium Carbide at 750 psi



2.00% Aluminum at 100 psi

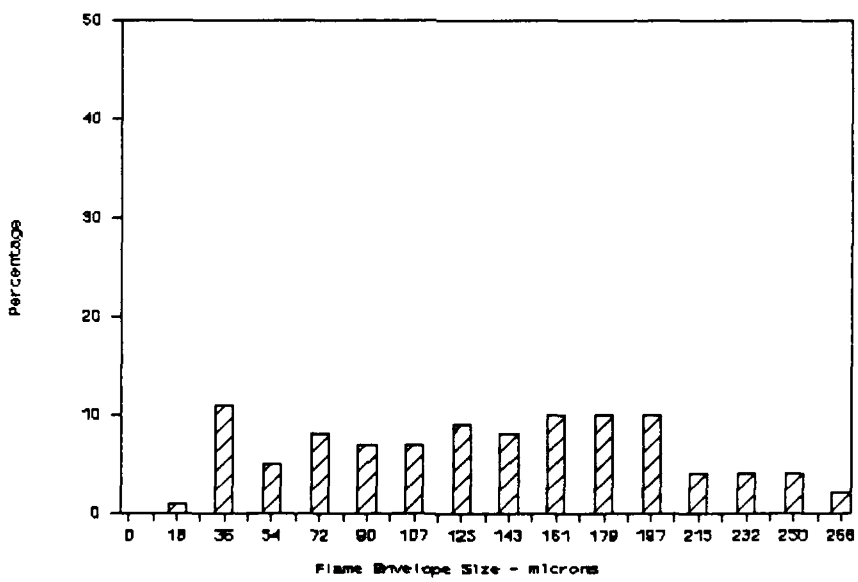
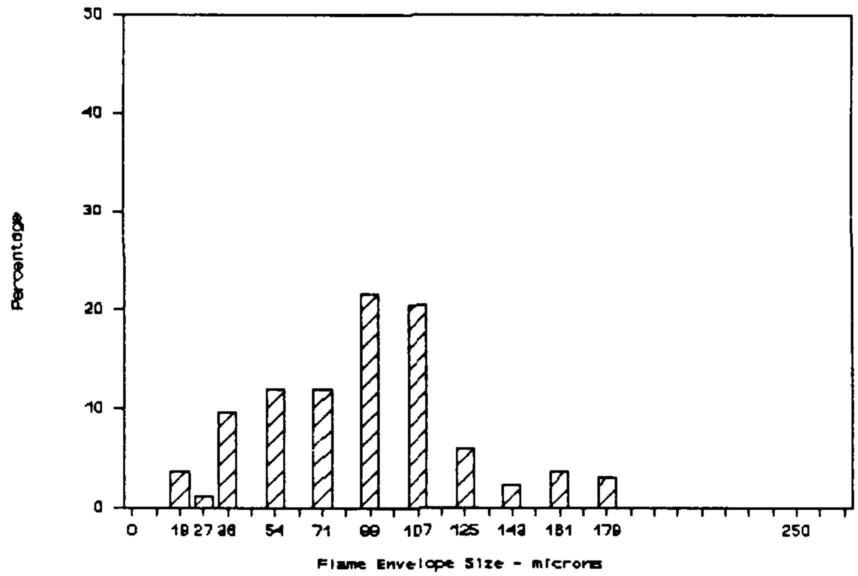


Figure 23. (CONTINUED)

2.00% Aluminum at 500 psi



2.00% Aluminum at 750 psi

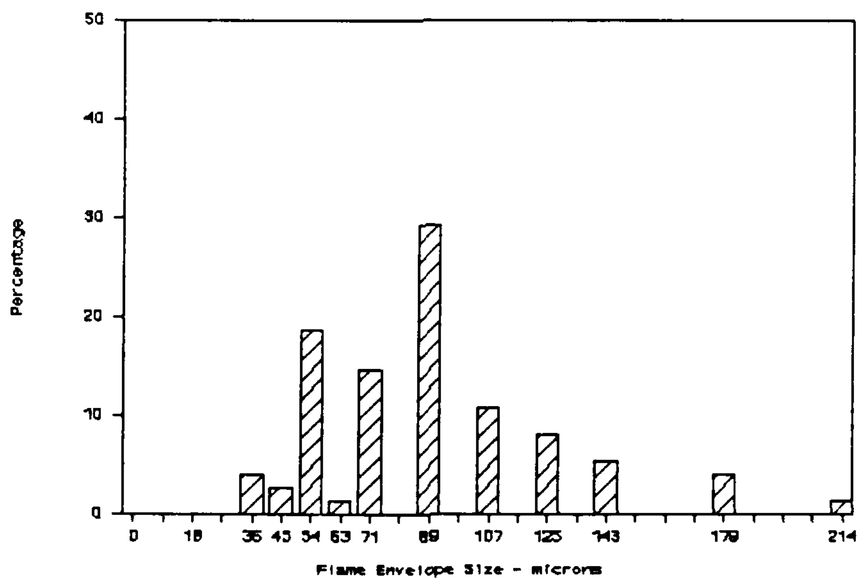
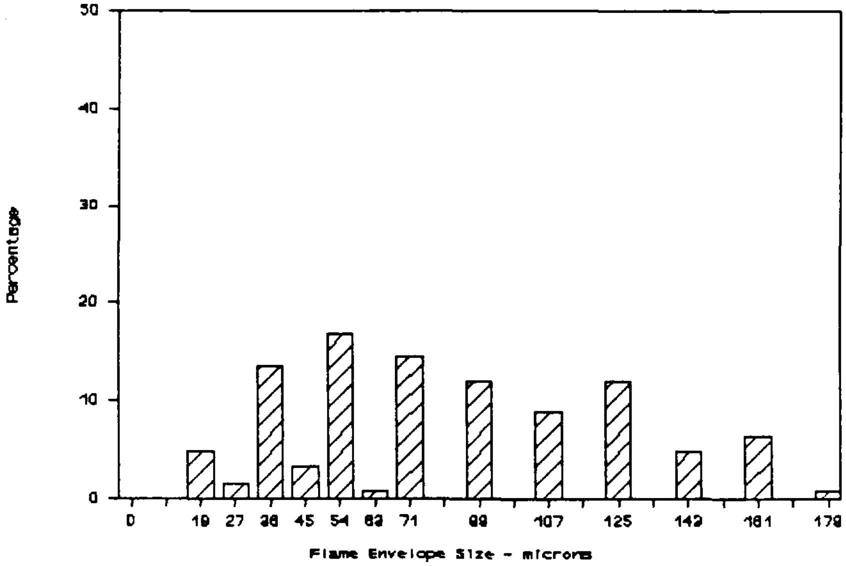


Figure 23. (CONTINUED)

4.68% Aluminum at 100 psi



4.68% Aluminum at 500 psi

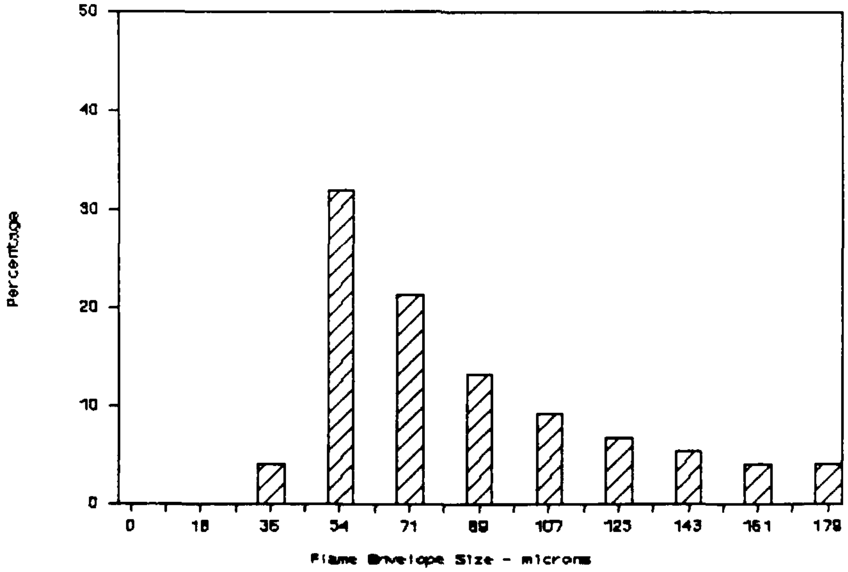


Figure 23. (CONTINUED)

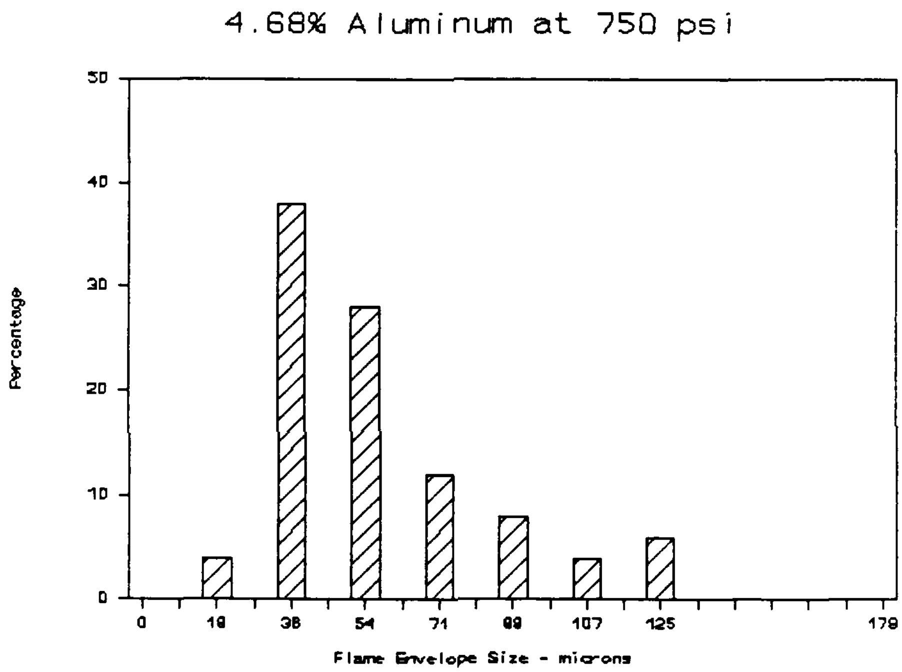


Figure 23. (CONTINUED)

Figure 24a. SEM Photograph of ZrC Residue at 100 Psi

Figure 24b. SEM Photograph of ZrC Residue at 500 Psi

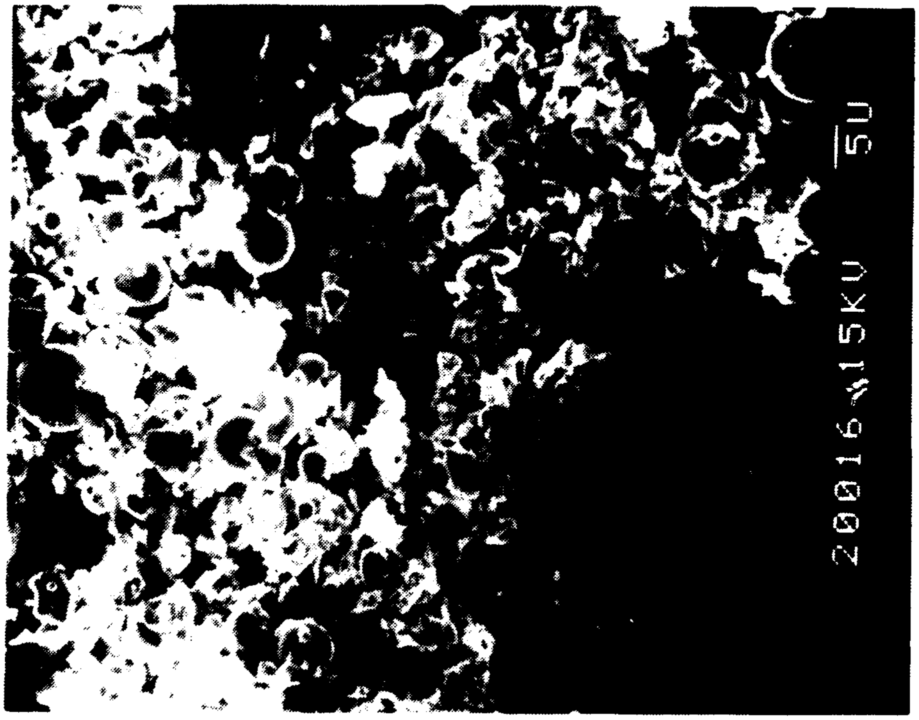


Figure 24c. SEM Photograph of ZrC Residue at 750 Psi

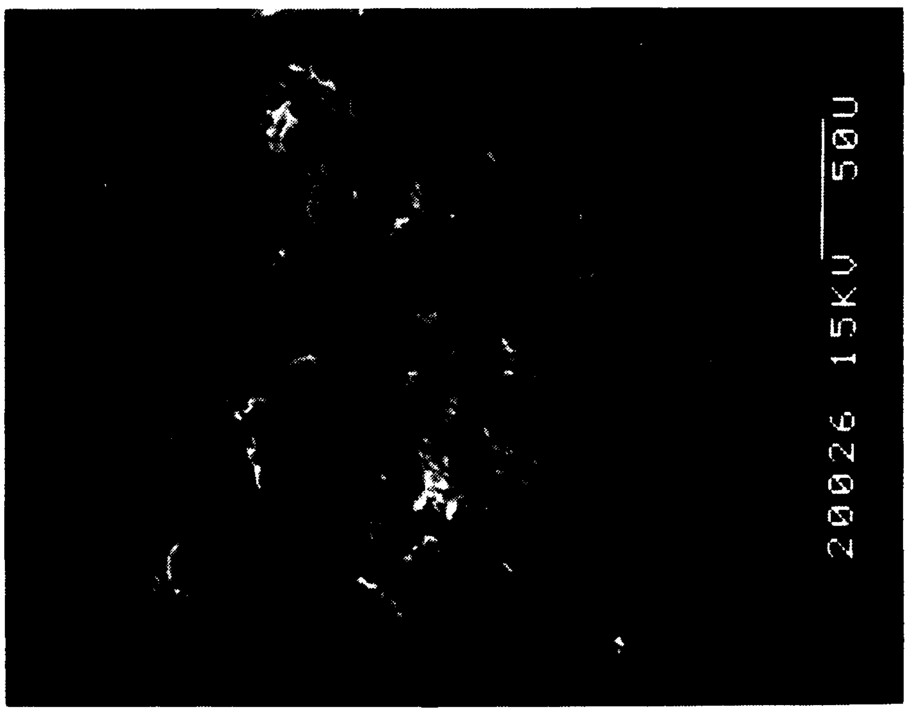


Figure 24d. SEM Photograph of 2.00% Al Residue at 100 Psi

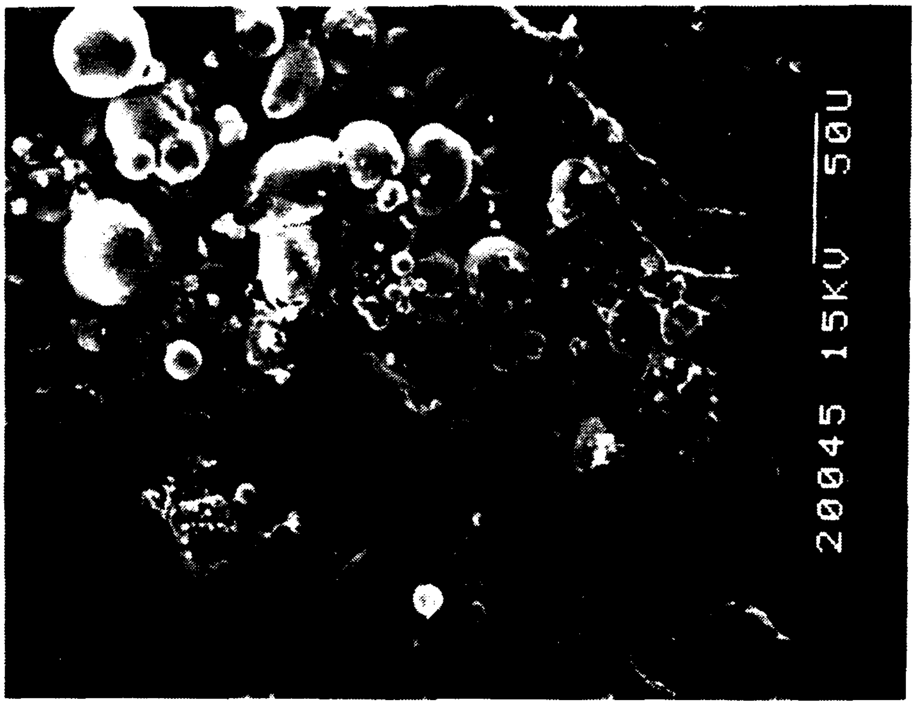


Figure 24e. SEM Photograph of 2.00% Al Residue at 500 Psi

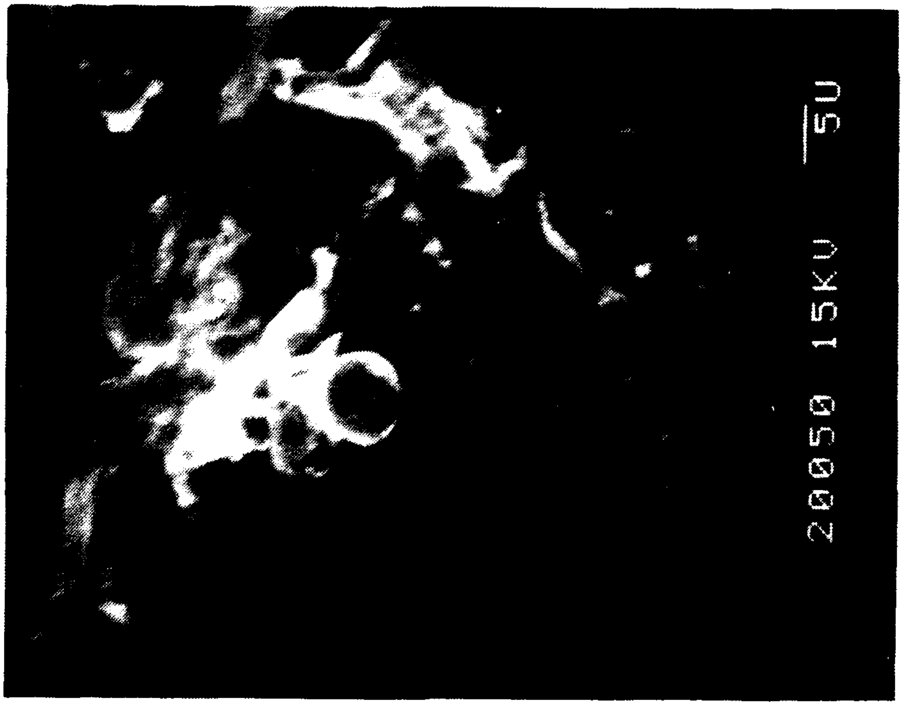


Figure 24f. SEM Photograph of 2.00% Al Residue at 750 Psi

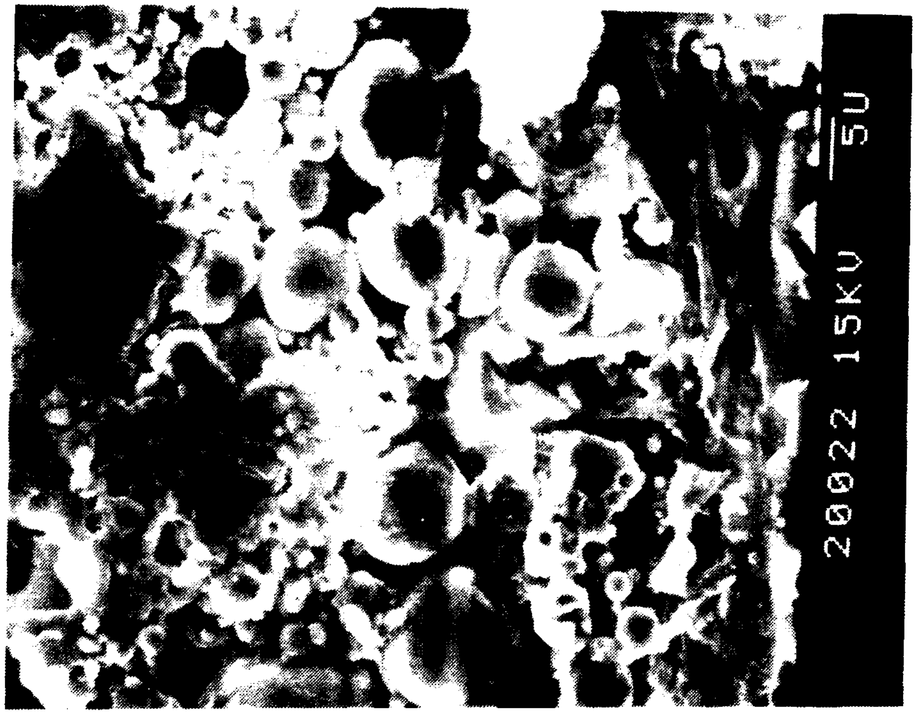


Figure 24g. SEM Photograph of 4.68% Al Residue at 100 Psi

Figure 24h. SEM Photograph of 4.68% Al Residue at 500 Psi

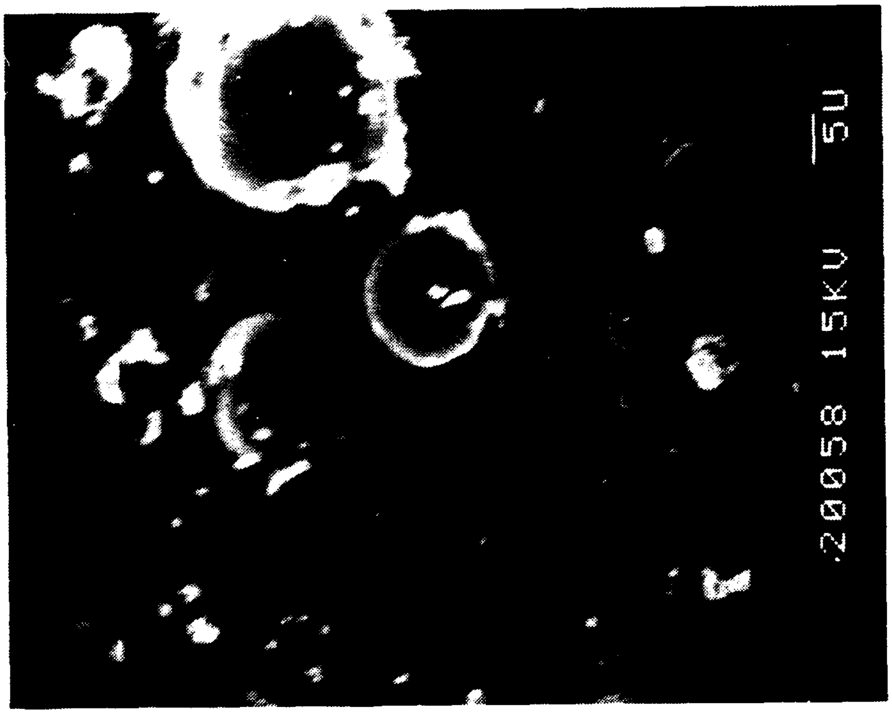
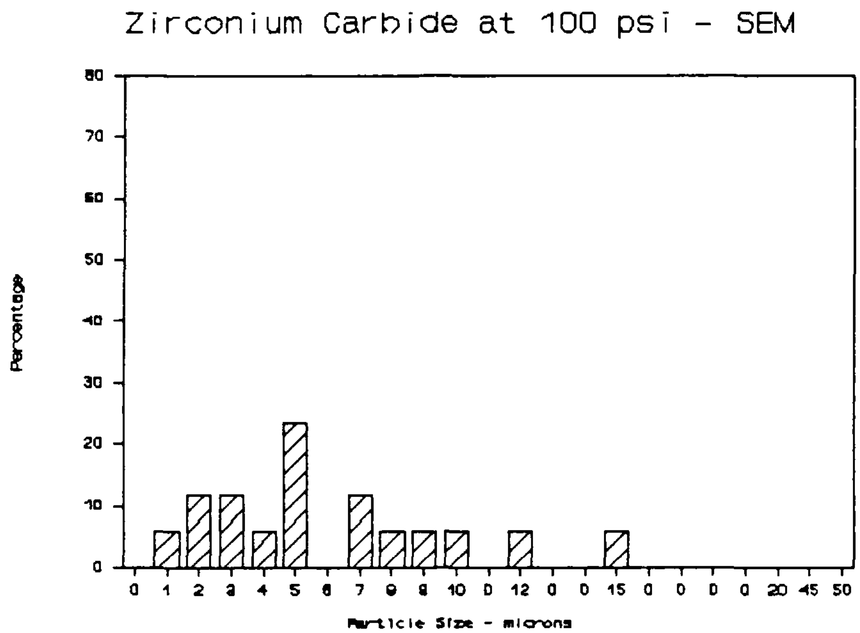


Figure 24i. SEM Photograph of 4.00% Al Residue at 750 Psi



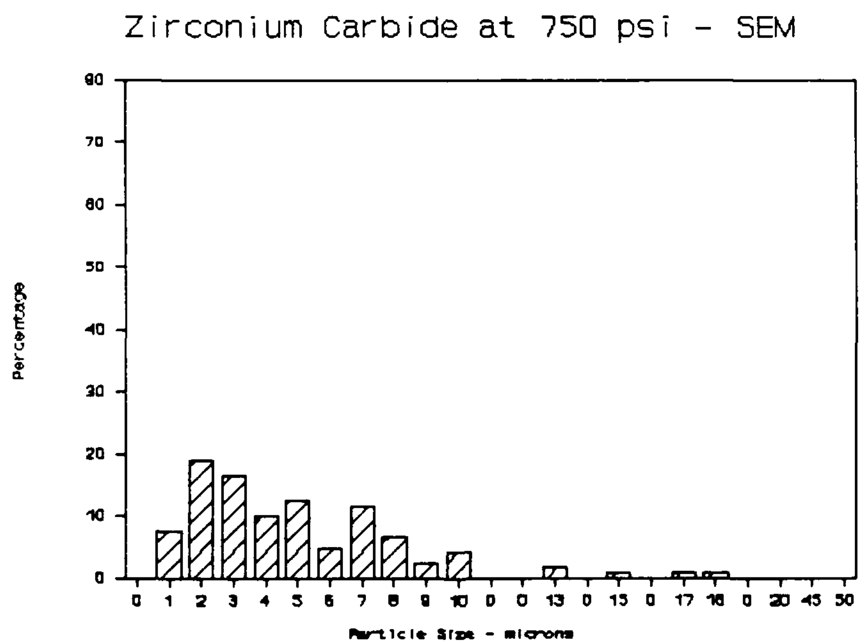
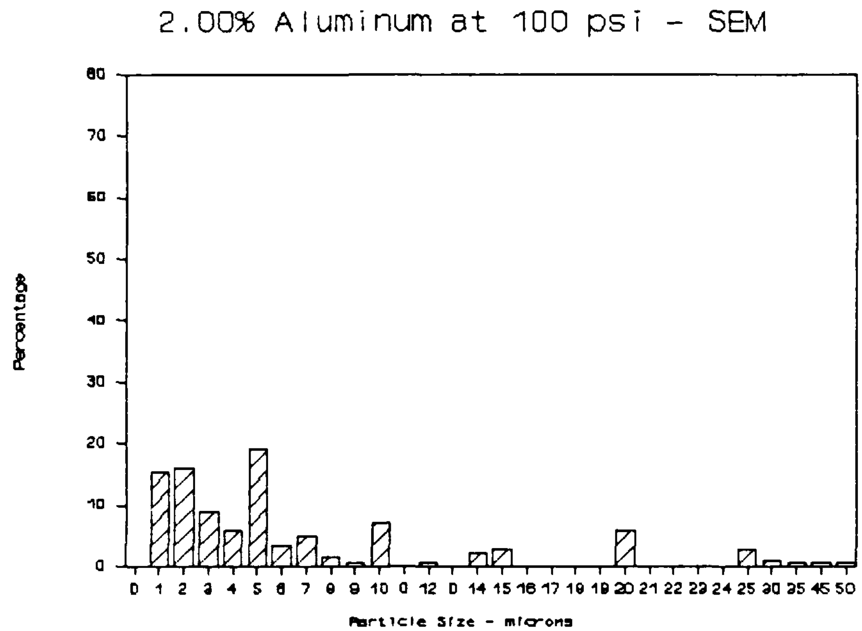


Figure 25. SEM Particle Sizes



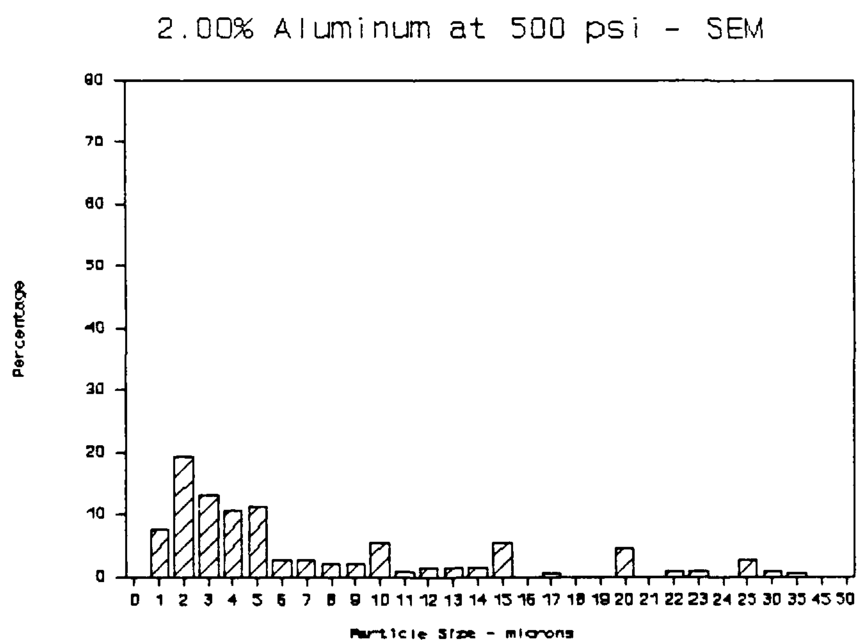
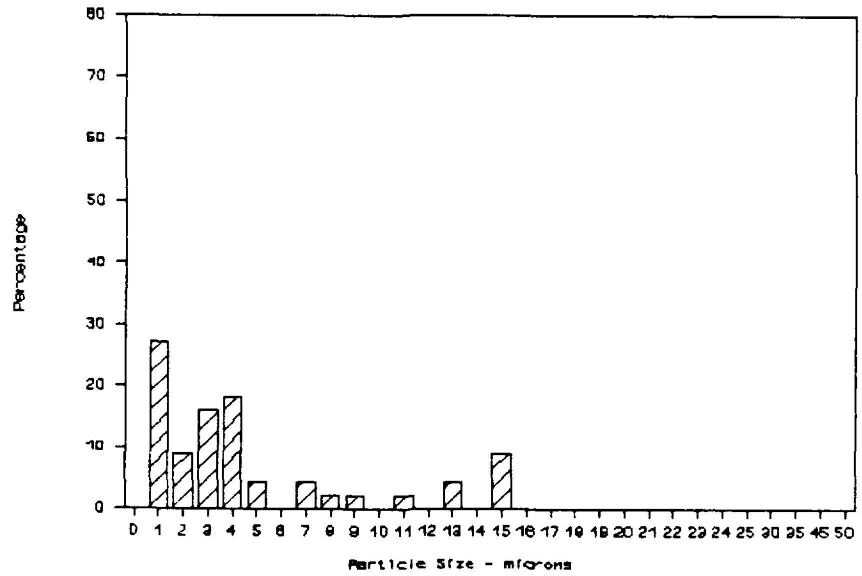


Figure 25. (CONTINUED)

2.00% Aluminum at 750 psi - SEM



4.68% Aluminum at 100 psi - SEM

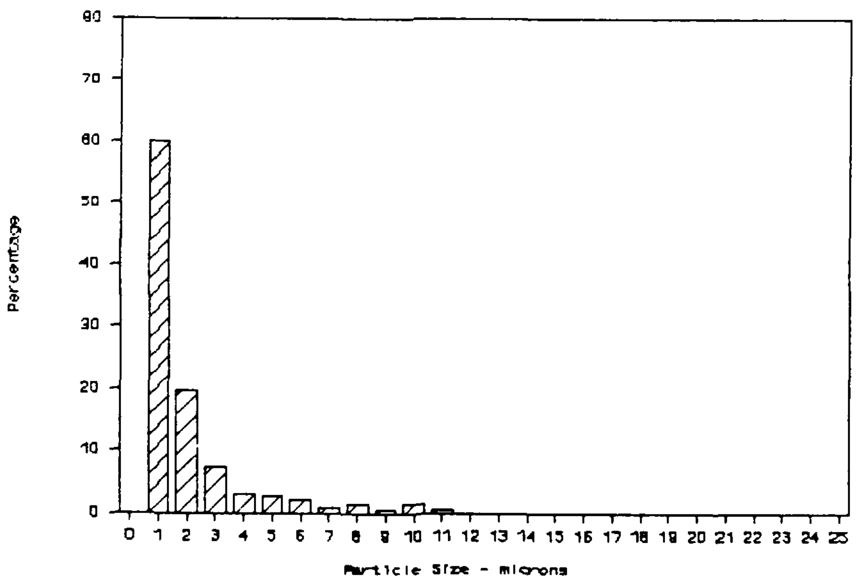
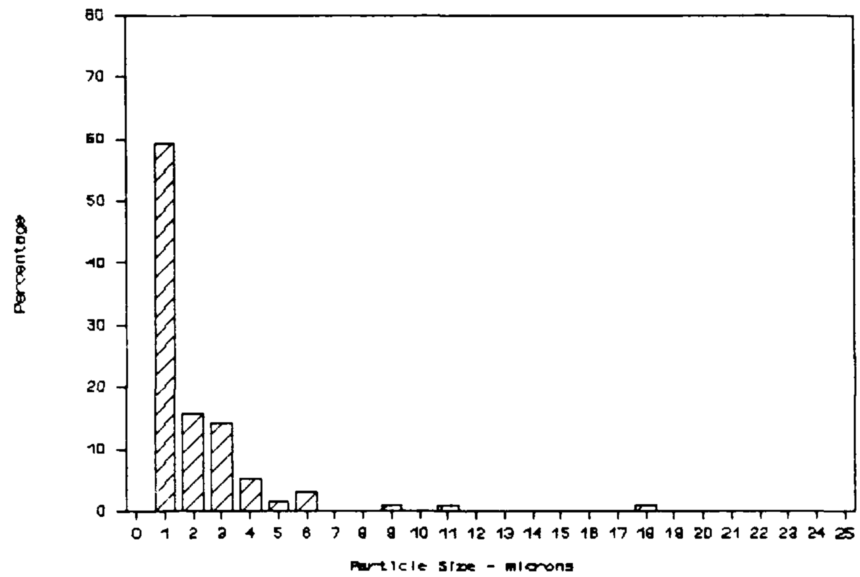


Figure 25. (CONTINUED)

4.68% Aluminum at 500 psi - SEM



4.68% Aluminum at 750 psi - SEM

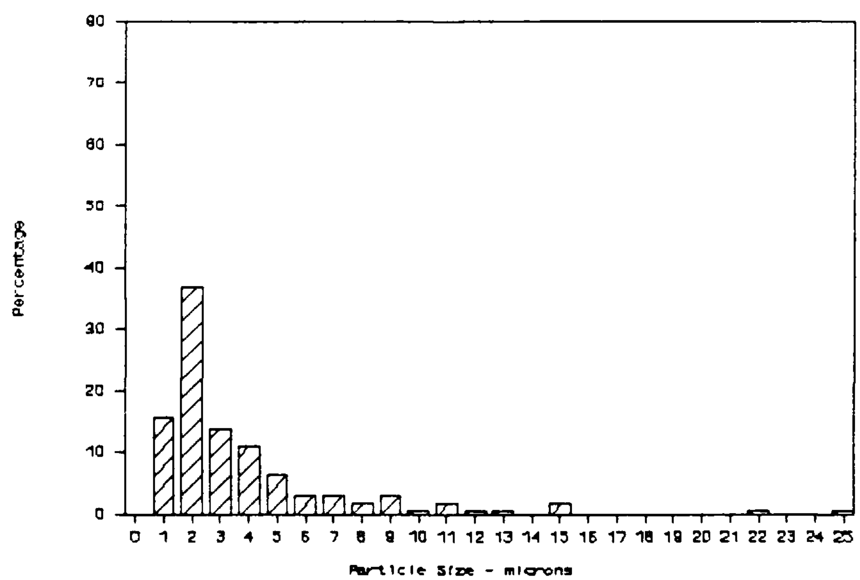
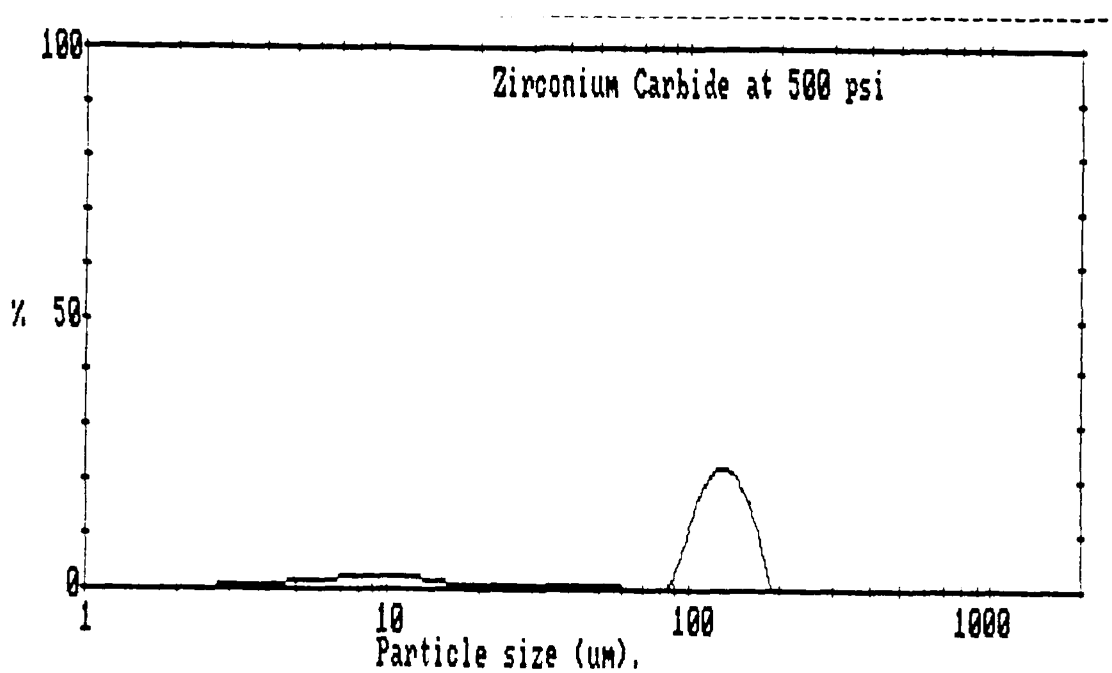


Figure 25. (CONTINUED)

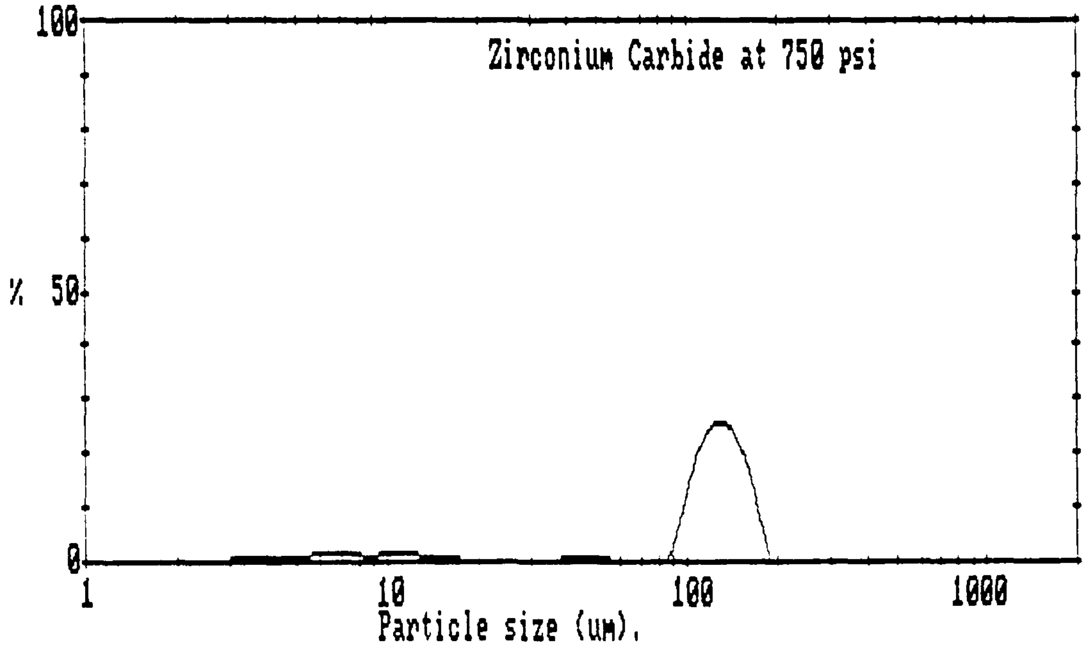


Malvern Instruments MASTER Particle Sizer M3.0 Date 30-11-88 Time 08-53

Size microns	% under	Size band microns		%	
					Result source=mike
					Record No. = 14
					Focal length = 100 mm.
188.0	100.0				Experiment type pil
87.2	25.0	188.0	87.2	75.0	Volume distribution
53.5	24.1	87.2	53.5	1.0	Beam length = 33.8 mm.
37.6	22.4	53.5	37.6	1.7	Obscuration =0.3329
28.1	21.5	37.6	28.1	0.9	Volume Conc. = 0.0107 %
21.5	20.4	28.1	21.5	1.1	Log. Diff. =4.56
16.7	19.0	21.5	16.7	1.4	Model indp
13.0	16.7	16.7	13.0	2.3	
10.1	12.7	13.0	10.1	3.9	D(v,0.5) = 119.0 µm
7.9	9.3	10.1	7.9	3.5	D(v,0.9) = 158.5 µm
6.2	6.1	7.9	6.2	3.2	D(v,0.1) = 8.3 µm
4.8	3.7	6.2	4.8	2.4	D(4,3) = 102.2 µm
3.8	2.2	4.8	3.8	1.6	D(3,2) = 25.0 µm
3.0	1.1	3.8	3.0	1.0	Span = 1.3
2.4	0.6	3.0	2.4	0.5	Spec. surf. area
1.9	0.3	2.4	1.9	0.3	0.05 sq.m./cc.

Sample details:-Zirconium Carbide at 500 psi

Figure 26a. Malvern Data of ZrC Residue at 500 Psi

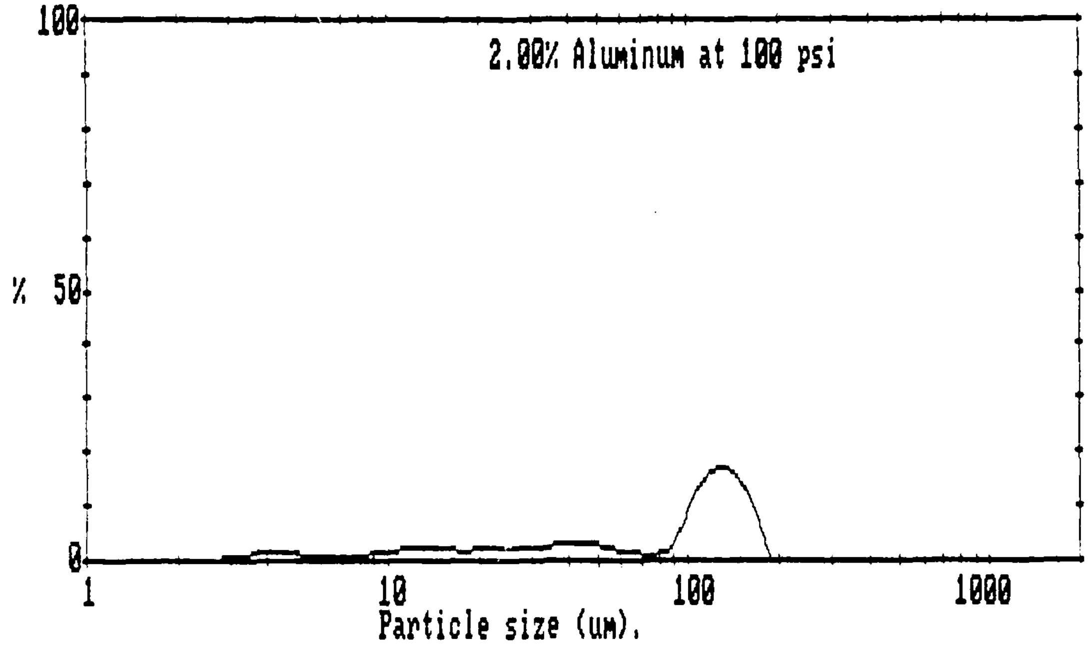


Malvern Instruments MASTER Particle Sizer M3.0 Date 30-11-88 Time 09-05

Size microns	% under	Size band microns		%	
					Result source=mike
					Record No. = 17
					Focal length = 100 mm.
188.0	100.0				Experiment type pil
87.2	14.8	188.0	87.2	85.2	Volume distribution
53.5	14.5	87.2	53.5	0.3	Beam length = 33.8 mm.
37.6	13.0	53.5	37.6	1.5	Obscuration =0.2506
28.1	12.8	37.6	28.1	0.2	Volume Conc. = 0.0105 %
21.5	12.6	28.1	21.5	0.3	Log. Diff. =5.18
16.7	12.0	21.5	16.7	0.6	Model indp
13.0	10.9	16.7	13.0	1.1	
10.1	8.6	13.0	10.1	2.3	D(v,0.5) = 124.2 µm
7.9	6.7	10.1	7.9	1.9	D(v,0.9) = 160.4 µm
6.2	4.5	7.9	6.2	2.2	D(v,0.1) = 11.8 µm
4.8	2.6	6.2	4.8	1.8	D(4,3) = 113.8 µm
3.8	1.5	4.8	3.8	1.1	D(3,2) = 31.0 µm
3.0	0.8	3.8	3.0	0.8	Span = 1.2
2.4	0.4	3.0	2.4	0.4	Spec. surf. area
1.9	0.2	2.4	1.9	0.2	0.05 sq.m./cc.

Sample details:-Zirconium Carbide at 750 psi

Figure 26b. Malvern Data of ZrC Residue at 750 Psi

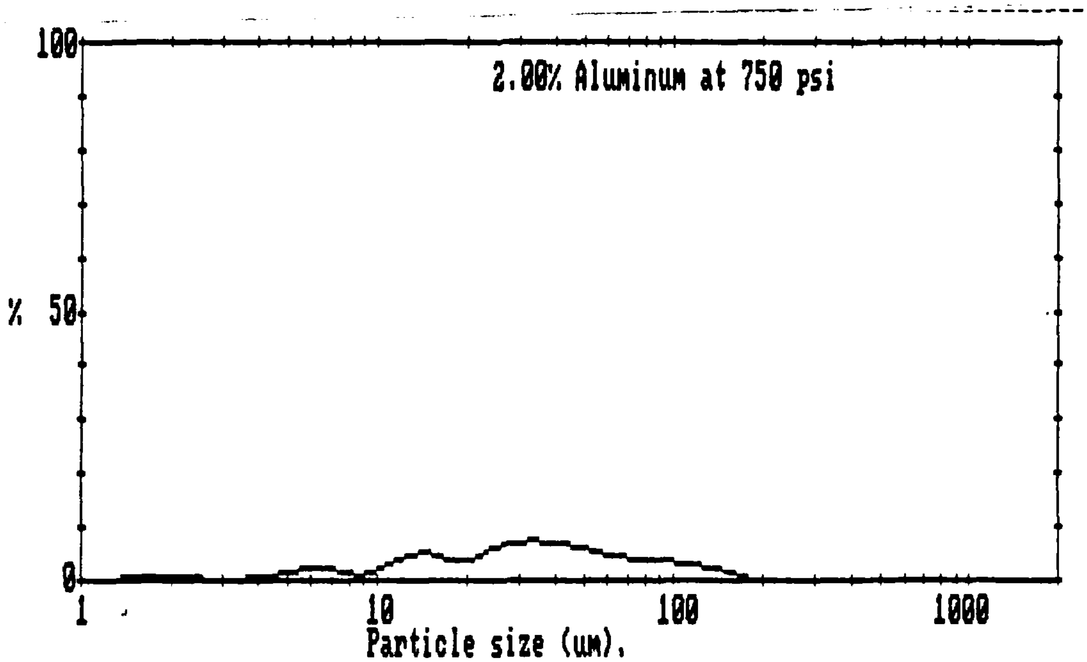


Malvern Instruments MASTER Particle Sizer M3.0 Date 30-11-88 Time 09-16

Size microns	% under	Size band microns		%	
					Result source=mike
					Record No. = 23
					Focal length = 100 mm.
188.0	100.0				Experiment type pil
87.2	42.4	188.0	87.2	57.6	Volume distribution
53.5	37.8	87.2	53.5	4.6	Beam length = 33.8 mm.
37.6	30.8	53.5	37.6	7.0	Obscuration =0.2137
28.1	25.8	37.6	28.1	5.0	Volume Conc. = 0.0051 %
21.5	21.5	28.1	21.5	4.3	Log. Diff. =4.46
16.7	18.3	21.5	16.7	3.3	Model indp
13.0	14.8	16.7	13.0	3.4	
10.1	11.4	13.0	10.1	3.4	D(v,0.5) = 104.5 µm
7.9	9.4	10.1	7.9	2.1	D(v,0.9) = 153.7 µm
6.2	7.6	7.9	6.2	1.7	D(v,0.1) = 8.6 µm
4.8	5.9	6.2	4.8	1.7	D(4,3) = 86.2 µm
3.8	3.5	4.8	3.8	2.4	D(3,2) = 21.4 µm
3.0	1.9	3.8	3.0	1.6	Span = 1.4
2.4	1.4	3.0	2.4	0.5	Spec. surf. area
1.9	0.9	2.4	1.9	0.5	0.05 sq.m./cc.

Sample details:-2.00% Aluminum at 100 psi

Figure 26c. Malvern Data of 2.00% Al Residue at 100 Psi



Malvern Instruments MASTER Particle Sizer M3.0 Date 30-11-88 Time 09-30

Size microns	% under	Size band microns		%	Result source=mike
					Record No. = 37
					Focal length = 100 mm.
188.0	100.0				Experiment type pil
87.2	88.1	188.0	87.2	11.9	Volume distribution
53.5	73.8	87.2	53.5	14.2	Beam length = 33.8 mm.
37.6	58.8	53.5	37.6	15.1	Obscuration =0.2319
28.1	44.8	37.6	28.1	14.0	Volume Conc. = 0.0037 %
21.5	34.3	28.1	21.5	10.5	Log. Diff. =3.51
16.7	28.1	21.5	16.7	6.2	Model indp
13.0	19.8	16.7	13.0	8.3	
10.1	14.0	13.0	10.1	5.8	D(v,0.5) = 31.4 µm
7.9	11.9	10.1	7.9	2.1	D(v,0.9) = 94.5 µm
6.2	8.5	7.9	6.2	3.4	D(v,0.1) = 6.8 µm
4.8	5.4	6.2	4.8	3.1	D(4,3) = 41.6 µm
3.8	4.2	4.8	3.8	1.2	D(3,2) = 14.4 µm
3.0	3.7	3.8	3.0	0.5	Span = 2.8
2.4	3.2	3.0	2.4	0.5	Spec. surf. area
1.9	2.2	2.4	1.9	1.1	0.06 sq.m./cc.

Sample details:-2.00% Aluminum at 750 psi

Figure 26d. Malvern Data of 2.00% Al Residue at 750 Psi

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